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EXPLOSIBILITY OF ENERGETIC MATERIAL DUSTS

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Hazard Research Corporation performed an experimental program to investigate the effects of particle size, dust cloud concentration, and relative humidity on the minimum concentration for explosion, minimum ignition energy and minimum ignition temperature of M-1 and M-30 propellant and explosives.

Southwest Research Institute determined the explosibility of M-1 propellant dust as follows: (1) the minimum energy of electrostatic discharge, (2) the minimum explosive dust concentration and (3) the effects of moisture, solvent, temperature, and particle sizes.

The Energetics Materials Division, LCWSL, ARRADCOM collected dust samples from representative sites along explosive and propellant production lines at several Army Ammunition Plants and determined whether dust hazards existed. Dynamic flow characteristics for various dust concentrations were established.

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SUMMARY

As part of the U.S. Army Modernization and Expansion Program, the Energetics Systems Process Division of ARRADCOM initiated a diverse program to study the problem of dust explosion which is always a potential hazard in the manufacture of explosives and propellants. This program was carried out in three phases, the results of which are contained in separate reports (Refs. 1, 2 and 3). In order to present a comprehensive discussion of dust explosion hazards in Army Ammunition Plants, the results of all three phases of this program have been summarized in this report.

Hazard Research Corporation performed an experimental program to investigate the effects of particle size, dust cloud concentration and relative humidity on the minimum concentration for explosion, minimum ignition energy and minimum ignition temperature of M-1, M-30, Composition B and HMX propellants and explosives (Ref 1).

Analysis of the results revealed that the material most sensitive to thermal and electrical initiation sources was M-1. M-30 was the second most sensitive followed by Composition B and HMX. The minimum ignition energy was observed to increase with increasing particle size, test chamber size, relative humidity, and decreasing dust cloud concentration. Also, minimum ignition temperatures were found to increase with increasing particle size.

It was also observed that as the particle size of M-1 and M-30 decreased below the 74-micron range, an electrostatic charging phenomenon caused the particles to agglomerate. This resulted in an increase in the minimum concentration for explosion for the smaller particle size ranges.

The data generated on this program should be used by the design engineers for the development of new production facilities as well as for the evaluation of existing production plants. An outgrowth of this effort could be the development of monitoring devices that sense dispersed dust clouds, compare concentrations to those required for ignition, and take appropriate action if a hazardous condition exists.

Southwest Research Institute was asked to study the explosibility of M-1 propellant dust (Ref 2), using a cursory set of experiments to determine: (1) the minimum energy of electrostatic discharge to induce an explosion, (2) the minimum explosive dust concentration, and (3) the effect of various

moisture, solvent, temperature and particle sizes have upon the explosion threshold values of each of three M-1 propellant concentrations.

The results of this exploratory study of M-1 propellant dust explosibility demonstrated most definitively that severe flash fires and explosions can indeed be initiated under certain sets of ambient conditions. Because of the exploratory nature of this study, it was possible only to identify the parameters which contribute to making an M-1 dust fire susceptible to an explosive reaction.

A detailed description of the test equipment and techniques used to determine the explosibility of M-1 propellant dust is given. Conclusions are drawn from the test program with regard to the minimum ignition energy and the minimum explosive concentrations of M-1 dust, and recommendations are made for expanding this exploratory study into a more detailed evaluation. Based on these exploratory tests in conjunction with the additional suggested tests, further recommendations should be submitted in reference to: (1) limitations of plant exposure to potential dust explosions, (2) suggested venting for dryers and operating areas, and (3) the proper design of a water deluge to combat any secondary fires resulting from a dust explosion.

Energetic Materials Div at ARRADCOM planned and executed a project to collect dust samples from representative sites along the explosives and propellants production lines at several ordnance plants (Ref 3). Air pumps and PVC filters were used. Except for some accumulation of dust in collectors and around milling operations, which could produce hazardous concentrations if stirred up by an external event, the sampling did not reveal any obvious explosive dust hazards. Data on ignition due to fire and shock will be required to more fully evaluate the worst case.

Energetic Materials Div also studied the explosibility of explosive and propellant dusts. A flow system was constructed which was specially designed to obtain more detailed data on the effect of particle size and concentration on the spark ignition of propellant and explosive dusts. Streak photography was employed to determine the velocity of flames and/or explosion waves. The ignition of M-1 propellants, RDX, HMX and Ball Powder was determined. The results were essentially in agreement with other studies where only the overall dust concentration in a closed vessel, not the local concentration near the ignitor, was characterized. Further comparison of the results of these tests to those of Hartmann Apparatus would be very helpful in establishing standards for hazard analysis.

INTRODUCTION

Background

Although dust explosions have been a recognized hazard for about 200 years, it has been mainly in the last 25 years in which the propellant and explosive technology has strived to provide the most efficient safety methods for the in-process operations. This attention to increased safety and efficiency has been addressed in detail as part of the U.S. Army's Production Base Expansion and Modernization Program which provides for construction of new explosive and production facilities, and the renovation and modernization of older facilities. In the interest of safety, many problem areas and techniques have been identified as potentially hazardous. Investigations were initiated to find means of reducing these conditions while maintaining minimal plant cost and maximum production efficiency.

In the manufacture of explosives and propellants there is always a hazard of dust explosion, especially in the drying lines where dust concentration is relatively high. The problem is compounded by the presence of varying amounts of water, ethyl alcohol, anhydrous ether in vapor form and in dust particles. An accidental electrical spark could ignite the dust/solvent vapor ridden air. If a detonation resulted, the main body of explosives or propellant would also detonate sympathetically, allowing no time for a water deluge system to respond, thereby producing disastrous results.

The introduction of the concept of "continuous" vs. "batch" production of sensitive materials has resulted in a marked increase in the requirement for dust control equipment. Higher production rates require the use of conveyor belts, fluidized bed dryers, continuous casting belts, automatic weighing and bagging machines, vibratory and weigh feeders. Proper design of dust control equipment requires a knowledge of the particle size distribution of the material being processed and the effects environmental conditions have on the dust explosion characteristics of the material being produced.

Hazard analysis requirements of ARMCOM Regulation No. 385-4 specify that all hazards be classified. This classification has been based on a comparison of the mechanical, electrical and thermal input energies that exist, due to normal process operations, with the sensitivity to initiation of the explosive material (threshold initiation level). Extensive investigation into the literature failed to uncover adequate data on the

ignition sensitivities of various forms of the materials selected for evaluation. Furthermore, the existing data banks did not provide detailed information on the effects of particle size, concentration, air temperature and relative humidity on the ignition sensitivities of these materials.

Realizing the potential hazard of dust explosion in the manufacture of explosives and propellants, the Energetics Systems Process Div of ARRADCOM initiated a three-phase program, the specific phase results of which are combined here to present a comprehensive report on the effects which produce dust explosions. These studies (or phases) were designated as:

1. Phase I - Dust Explosion Sensitivity Tests on M-1, M-30, Composition B and HMX.
2. Phase II - Exploratory Study of M-1 Propellant Dust Explosibility.
3. Phase III - Explosibility of Explosive and Propellant Dusts.

In addition to the above, a general discussion on dust explosion is presented to help the reader understand the nature of the problem.

Objectives

The objectives of the program in each of the three phases are as follows:

Phase I Objectives

Hazard Research Corporation initiated this phase of the program to investigate the effects of particle size, dust cloud concentration and relative humidity of the process air environment on the dust explosion characteristics of HMX, Composition B, M-1 and M-30 explosives and propellants. The program was divided in three parts to meet the following objectives:

Part A Objectives

1. Determine the effect of particle size on the minimum concentration for explosion at ambient air conditions.

2. Determine the effect of particle size on the minimum spark ignition energy under ambient air conditions at five times the minimum concentration for explosion.
3. Determine the effect of particle size on the minimum ignition temperatures of HMX, Composition B, M-1 and M-30 dust clouds.

Part B Objectives

Determine the effect of temperature, particle size and relative humidity on the minimum ignition energy of the energetic materials at five times their minimum concentration for explosion.

Part C Objectives

Determine the effect that reduced dust loadings have on the minimum spark ignition energies obtained in Part B. Evaluate these energies for various particle sizes and dust concentrations of three and one times the minimum concentration for explosion of the respective materials.

Phase II Objectives

Southwest Research Institute was charged to concentrate on the explosibility of M-1 propellant dust in this phase of the program. The objective was to determine the following parameters through a brief cursory set of experiments:

1. Minimum energy of electrostatic discharge to induce an explosion.
2. Minimum explosive dust concentration.
3. The effect of three different values of moisture, solvent temperature and particle size on the detonation threshold value of three M-1 propellant dust concentrations.

Phase III Objectives

This phase of the program was carried out by the Energetic Materials Div of ARRADCOM in two parts to meet the following objectives:

1. Collect dust samples from the production lines of explosives and propellants at various Army Ammunition Plants to gain a firsthand knowledge of the qualities and characteristics of the dusts generated.
2. Measure the minimum dust concentrations of various materials required to start an ignition with a resultant flame propagating from the ignition.

DISCUSSION OF DUST EXPLOSION

A general definition of dust is "a collection of finely divided solid particles".

Any combustible solid when divided into minute particles and then suspended in air can explode. Such seemingly innocuous substances as starch, dusts from grain, bark, cotton and wood, when finely divided and dispersed into the air, can become extremely dangerous (Ref 4). One source (Ref 5) states that the number of major dust explosions in the U.S. alone between 1900 and 1959, excluding coal mine explosions, was 1,100. A total of 648 people were killed in these accidents. Also, a propellant plant exploded in The Netherlands in which two people were killed. This explosion was attributed to the ignition of a layer of propellant dust by static electricity (Ref 6). As recently as December 1977, grain dust explosions in Louisiana and Texas have caused tremendous damage and loss of human life.

Dust can be classified as:

1. Capable of ignition with a resultant flame propagation from the ignition, and
2. Capable of ignition but without any flame propagation. It is only those that fall into Category 1 that constitute a hazard. Sources that produce the largest number of dust particles are those in which solids are undergoing an energetic process such as grinding, conveying, mixing or impacting other solid bodies in some fashion. In some of these processes, dust is the desired product; while in others, it is an unavoidable consequence. In any case, dust particles can be dispersed from their source in one of three ways. One is dynamic protection. Despite tremendously high initial velocities, calculations do reveal that dust particles will travel only a short distance. Another mode of dispersion is diffusion, which is relatively slow. The third and primary mode of dispersion is transport by air currents. This fact is important in controlling dust levels around dust sources (Ref 7).

The increased chemical activity of dusts can be largely attributed to the greatly increased surface area per unit mass (Ref 8). The increased surface area allows more oxygen per unit mass to be absorbed, which allows the particle to burn more rapidly when ignited. Dust behaves in some ways like a fluid,

and, therefore, with reservation could be termed a "discrete" fluid. Dust can be poured like a liquid, compressed and evaporated by passing air through it.

There are many parameters associated with dust and dispersed dust (dust clouds). Dust parameters can be classified into two groups: those parameters which refer to individual particles and those which refer statistically to the whole dust particle population. One important parameter is particle size. However, particle size cannot be specified without taking into account the shape of the particle. Many criteria have been developed for specification of particle size of irregularly shaped particles. For the purposes of this investigation, particle size is determined by sieving. It is sometimes useful to determine the particle size to mass distribution of a dust sample. When dust is dispersed into the air, a useful parameter is the number of particles per unit volume; i.e., particle concentration. A more convenient and more generally used equivalent expression is dust mass per unit volume, or dust mass density. The two expressions are interchangeable if the particle size to mass distribution is known. When expressing a dust concentration, it must be realized that a dust cloud consists of discrete solid particles; therefore, a dust concentration loses meaning in the limit of small volumes. This is important to realize, for example, if the device used to ignite a dust cloud is very small, such as an electrostatic spark.

In the definition above for dust, no ranges were specified for particle size. Yet, there should be particle size transition from molecular to "dust" to "ordinary" particles. One method of classification of particles by size is based on the terminal velocity at which the dispersed particles will fall back to earth. Generally, the smaller the particle, the lower the terminal velocity. In a dust cloud with a distribution of particle sizes, the mean dust particle size would decrease with time as the larger particles would settle out, providing particle replenishment does not occur. One group of particles [under 1 micron (9.906×10^{-5} cm) in size] behave like a gas when suspended in air. The terminal velocities are very low (<1 cm/sec) and Brownian motion masks the downward settling motion. A second group of particles lies in the range of about 1 to 120 microns (9.906×10^{-5} cm to 1.189×10^{-2} cm). The terminal velocity of these particles is fairly accurately predicted by Stokes Law and its upper limit is about 30 cm/sec. All particles above 120 microns form the last group with terminal velocities increasing with particle size in a complex fashion (Ref 10). It has been reported by Magison in Reference 11 that the largest particles that actively contribute to a dust explosion were 380

microns (3.76×10^{-2} cm) in size. Therefore, for the purpose of this report, reference to dust will imply particle sizes of 380 microns or less.

Two conditions are necessary for a dust cloud to explode. The dust concentration must lie between certain limits and sufficient energy must be supplied to initiate the reaction. "Dust explosibility" is the relative ease with which a dust cloud will ignite and explode. Many parameters affect the explosibility of dust: concentration, particle size, air temperature, moisture content of the dust particles, and the presence of foreign particles or vapors. Most important for industrial operations is the determination of minimum ignition energy of a dust cloud and minimum explosive concentration. Minimum explosive concentrations vary from 10 to 600 mg/l (Ref 12). It should be noted that at a concentration of 50 mg/l, the minimum explosive concentration for wheat flour is equivalent to 1 gm per 0.028 m³. Breathing air containing dust at this concentration would be difficult and visibility would be severely limited. A good rule of thumb is that if one can see his outstretched hand, the dust concentration is probably below the minimum explosive concentration (Ref 13). Listed below are some commonly encountered particle concentrations in mg/l (oz/ft³ x 10⁻³) (Refs. 14 and 15).

<u>Concentration</u>	<u>Significance</u>
10 - 5,000	Explosive Concentration of Dusts
.4 - .7	Dust Storm
.02 - .3	Mine Air
.008 - .03	Fog, Mist
.0002 - .007	Industrial District Air
.00007 - .0007	Rural and Suburban Air

It can be seen that explosive concentrations of dust are not found in most situations and are probably restricted to the immediate area around intense sources of dust particles.

Spark Ignition

Probably the most likely ignition source to be encountered in practice is a spark discharge. Sparks are known to be effective ignition sources and can be well controlled and reproduced in the laboratory. Thus, spark ignition was chosen for this study. The goal was to measure a minimum concentration of dust at which ignition and flame propagation will occur for a particular energetic material. The manner in which ignition

energy varies with dust concentration provides a basis for the minimum dust concentration concept.

Figure 1 shows a typical ignition energy-dust concentration curve. The abscissa and ordinate axes in Figures 1 and 2 are not assigned a specific numerical scale. The purpose of the two curves is to show the relative position of values encountered in dust explosibility work. It may be noted that, in the region of minimum explosive concentration, the required ignition energy is a rapidly varying function. This fact is very important in the determination of minimum ignition energy and minimum explosive concentration. Also note that, in the transition zone, the dust burns but does not satisfy criteria for detonation. Figure 2 shows the anticipated ignition energy-dust concentration curve for M-1 propellant dust. Because M-1 dust does not require oxygen, the upper explosive concentration limit will be well beyond the value (for example, 2-5 gm/l for coal dust) for conventional dusts. This fact suggests that propellant dusts could supply insight into the mechanisms of dust explosions, such as attempting to explode a propellant in an inert atmosphere. Also, it should be noted that propellant dust in the propellant plant environment might have large quantities of ethyl alcohol or anhydrous ether with it, which would surely affect the dust explosibility.

As stated earlier, for a dust explosion to occur, there must be supplied a certain minimum ignition energy. There are many ways to introduce the necessary ignition energy into a dust cloud. One way is with an open flame. Other ways are frictional heating or sparks, intense radiation, or even shock waves from a nearby explosion. Finally, there is the electrical discharge spark. A static electrical discharge is the most likely ignition source in an M-1 plant. Minimum ignition energies in the form of electrostatic discharge are as low as five millijoules for some dusts. Some electrical spark energies and their significance are listed in Table 1. From this table it can be seen that relatively small amounts of energy are required to ignite a dust cloud, and that the energy produced from a human induced spark is sufficient in magnitude.

Previous studies that attempted to measure the minimum ignition energy have had difficulties in obtaining unambiguous data. For example, studies of spark ignition of gaseous methane air resulted in a minimum spark energy (Refs. 21 through 27) of 0.28×10^{-3} joule for an 8.5 percent methane air mixture. Priede (Ref. 28) then demonstrated that a series resistance of 5×10^4 ohms introduced in the capacitive discharge circuit reduced the minimum energy to about half of that obtained without the

resistance. Based on this finding he then postulated that the resistance lengthened the spark time which leads to a reduction in energy losses from heat conduction (Ref. 29).

A similar situation exists in determining the minimum energy for dusts. Line (Ref. 30) employing a high speed camera showed that sparks can disturb the dust cloud adjacent to the spark. In fact, his photographs demonstrated the existence of a small dust-free region adjacent to the spark. Eckhoff found a similar result (Refs. 31 and 32). The studies by Boyle and Llewellyn (Ref. 33), Priede (Ref. 28) and Moore (Ref. 34) have shown that a series resistance in the spark discharge circuit can dramatically affect the minimum energy for ignition. Studies using a wide variety of spark circuits have confirmed that long duration sparks will be more efficient in producing ignition than short sparks (Ref. 35). Eckhoff postulated that the details of how the spark disturbs the dust cloud cause this effect (Refs. 29 and 32).

The value of C_{min} is not so apparatus-dependent. Since the slope of the curve becomes very large, changes in the ignition energy do not produce minimum explosive concentrations that differ noticeably from C_{min} . Thus, measurements of C_{min} can be made by choosing a sufficiently large ignition energy to insure operation on the nearly vertical section of the curve and simply varying the dust concentration until no ignition is obtained. This has been the approach adopted in Phase I.

A problem with closed bomb measurements of C_{min} is obtaining a uniform dust concentration throughout the test volume. Usually, a weighed amount of dust is dispersed in the closed bomb by a blast of air. Non-homogeneity of the dust within the bomb is not characterized, although, as pointed out by Eckhoff, concentration gradients are likely to occur. The spark apparatus designed for Phase I attempted to eliminate the problem by providing a steady flow of dusty air past the electrodes. Dust concentrations were determined through measurements of dust collected in a given period of time.

Types of Explosions

Dust in which a reaction propagates by the combustion of particles with the suspending gas is said to be "explosible". Explosives and monopropellant dust do not require any such external gaseous oxidizer to undergo reaction; therefore, such dust in an inert gaseous medium may explode. However, in practice, it is airborne dusts that are important and atmospheric oxygen which may be involved in the explosion. Thus, all tests

conducted in Phase I examine the explosive property of these dusts.

Within the definition of the general term "explosion", there are two processes to be considered: deflagration and detonation. A deflagration is a flame propagating through the dust at velocities less than the local speed of sound. Pressure waves associated with this process are typically mild and overpressures developed in an unconfined environment are less than one atmosphere. Conversely, a detonation is a reaction propagation through the dust at velocities much greater than the speed of sound. Strong shock waves are associated with such an event. Some dusts will support both types of processes depending on the details of the ignition. Strauss (Ref. 36) has shown that aluminum dust in oxygen will detonate, while Lu (Ref 37) has demonstrated detonation in aluminum dust-air mixtures. Another study has shown that coal dust-air mixtures can be made detonable if they are sensitized by addition of methane (Ref. 38). A detonation process is much more destructive and far-reaching than a deflagration in the same dust.

Deflagrations in some materials can undergo transition to detonation. Generally, this transition requires some sort of confinement or obstacle within the volume of explosive material. Here, the deflagration of part of the explosive mixture sets up a flow of the unreacted mixture along the obstacle or confinement. Turbulence is generated in the flow ahead of the flame. When the flame interacts with this turbulence, a localized auto-explosion may occur, starting the detonation, then consuming the remainder of the explosive mixture. What started as a slow deflagration becomes a detonation producing a strong blast wave. The small region where the auto-explosion occurs is called an "exothermic center". Currently, it is not possible to accurately prescribe the conditions leading to the formation of these centers, although some materials are much more likely to form them than others. A dust's potential to undergo transition should be evaluated along with its potential to become ignited and propagate a flame.

The preceding paragraphs have provided some insight into the phenomena of dust explosions. In planning the experimental test program, a host of references were examined, two of which were deemed to be the most valuable and were used extensively throughout the test program. K.N. Palmer's book entitled Dust, Explosions and Fires (Ref 39) surveys the research that has been carried out on explosive hazards and also deals with industrial processes and preventive measures for safe handling of dust. Of greater importance is the Bureau of Mines Report No. 5624 (Ref.

40) which details the accepted procedures for the use of the Hartmann Apparatus for determining the minimum energies and minimum dust concentrations which could result in a potentially explosive situation. The Bureau of Mines procedures for determining dust explosibility were followed as closely as possible in the research program to be described later in this report.

PHASE I - DUST EXPLOSION SENSITIVITY TESTS ON M-1, M-30,
COMPOSITION B AND HMX

Materials

The following materials were supplied by ARRADCOM for use in this phase of the test program:

1. HMX, Grade B, Classes 1, 3 and 5, Lots GHBC-4, 6HG5-3, 6HCC-11A2
2. Composition B, Grade A, Spec. Mil C 0040 A, Lot HUL-053-5068 (screened)
3. M-30 Propellant, Mil P-46458, Lot RAD 67595 (Class 2)
4. M-1 Propellant.

Equipment

ARRADCOM supplied the following equipment:

Aqua Test II, Model #702 by Photo Volt Corporation, S/N 1095 (automatic Karl Fischer titrator).

Hazards Research Corporation (HRC) supplied the following equipment:

1. Hartmann Explosibility Apparatus
2. Godbert-Greenwald Furnace
3. Moulinex grinder
4. Tyler Sieve Shaker
5. U.S. Sieves, No. 100 (149-micron opening); No. 200 (74-micron opening); and No. 325 (44-micron opening).

Test Procedures

Sample Preparation

Composition B, M-1 and M-30 samples that satisfied the through 100, 200 and 325 U.S. sieve size requirement were not readily available for use in this phase of the program. Consequently, HRC prepared the samples by remotely impact

grinding the dry materials. The samples were prepared in 20-gram batches. Each batch was made by subjecting the material to the grinding action from 20 to 60 seconds. Duration was the controlling parameter for particle size reduction. Final particle size classification was accomplished by sieving the sample through the appropriate U.S. series sieve. All samples were then stored in airtight containers for future testing.

HMX samples were tested as received from the Government. Three granulations were supplied, Classes 1, 3 and 5.

Military Specification H-45444 B (PA) contains the particle size distribution for these three classes of HMX. This data has been extracted from the specification and is presented in Table 2; while the U.S. standard sieve numbers and corresponding sieve openings are presented in Table 3 for future reference. It can be observed from Table 2 that Class 5 HMX contains the smallest particles followed by Classes 1 and 2 in order of increasing particle size.

Sample Volatiles Content

The volatiles content of each material has been determined by vacuum drying it for 6 hours at 60°C. Table 4 presents the results of the volatiles content determinations.

Test Air Environmental Control Methods

To meet the objectives of Part B, it was necessary to develop a technique for controlling the temperature and relative humidity of the air in the Hartmann apparatus. The goal was to subject each dust cloud to a specific environmental condition and to determine the effect this condition had on the minimum ignition energy. Listed as follows are the two air temperatures and the three corresponding relative humidities that were selected:

Test condition	Air temperature (°C)	Relative humidity (%)
1	24	2
2	24	50
3	24	76
4	52	2
5	52	39
6	52	58

Temperature Control

Air temperature inside of the air reservoir and the lucite air chamber was raised to either 24°C or 52°C using fiberglass-wrapped, nichrome-heating ribbon. Electrical power to the heating ribbon was controlled using a variable auto-transformer. Thermocouples placed inside of the air space of the air reservoir and lucite tube monitored their air temperatures. Heating ribbon was wrapped around the base of the dust dispersion cup to accelerate its heating rate. Adjustments were continuously made to the auto-transformer until the desired steady-state air temperature was achieved.

Relative Humidity Control

The relative humidities reported in this program were attained by syringing calculated amounts of water into the heated air reservoir and dust dispersion cup. After the water vaporized, the reservoir was discharged into the lucite tube. A sample of air from the tube was withdrawn, using a syringe, and analyzed for water content in the Photovolt Aquatest II, automatic Karl Fischer titrator. This procedure was repeated until the results were reproducible. Fifteen reproducible trials were performed at each temperature and relative humidity condition before the dust samples were introduced into the apparatus. Therefore, the environmental test air conditions used in Phase I represent experimentally measured quantities. The 2 percent relative humidity values were obtained by purging dry, bottled compressed air through the Hartmann Apparatus for two minutes prior to activating the "fire" switch.

Calibrated Environmental Air Conditions Test Procedure

The experiment was initiated by engaging the following equipment:

1. Air pressure (cylinder of dry air)
2. Power supply
3. Heating tapes
4. Potentiometer (thermocouple readout)
5. Photovolt - Aquatest II

Adjustments to the following items were then made:

1. Air pressure (69 kPa)
2. Power supply (adjust energy setting between 50 and 8,000 mJ)
3. Heating tapes (24°C or 52°C)
4. Potentiometer (read millivolt output of iron constantan thermocouples)
5. Photovolt-Aquatest II (ready to receive sample).

The following sequence of operations were performed for each test cycle:

1. Distilled water was syringed into the brass reservoir and pressurized to a final pressure of 69 kPa at the desired temperature.
2. Distilled water was syringed into the brass dust dispersion cup of the lucite Hartmann tube.
3. The lucite tube was secured and the high voltage leads were connected to the electrodes.
4. Fifteen minutes were allowed to elapse for vaporization of the water to occur.
5. The "fire" switch on the power supply was activated.
6. An air sample was removed from the Hartmann tube using a syringe. This sample was injected into the liquid solution of the automatic titrator. The parts per million of water in the air sample were read directly from the titrator.

Minimum Ignition Energy Tests Under Controlled Environmental Air Conditions

All tests conducted on the energetic materials followed the procedures described above. The only differences were the introduction of a weighed amount of explosive sample after the 15-minute vaporization period had elapsed and the elimination of the air sample removal (sixth item).

Description of Tests

Part A Tests

The experiments performed during this part of Phase I of the program utilized three standard types of laboratory equipment developed by the Bureau of Mines and the United States Department of Labor, namely:

1. Hartmann apparatus for determining the minimum spark ignition energy for ignition of a dust cloud.
2. Hartmann Apparatus for determining the minimum concentration for explosion of a dust cloud.
3. Codbert-Greenwald furnace for determining the minimum ignition temperature of a dust cloud.

Minimum Spark Ignition Energy of a Dust Cloud

The minimum electrical energy required to ignite a dust cloud is determined in the Hartmann Apparatus (Appendix A).

The energy of the spark (in joules) is calculated as $1/2 CV^2$ where C is the capacitance of the condensers in farads, and V is the charging potential in volts. Dust cloud minimum ignition energy is the least amount of energy required to produce flame propagation of 10 cm or longer in the tube. Four trials are made at each condenser setting; however, if the dust ignites in initial trials, lower energy is tried until a minimum is obtained. A typical dust explosion in the lucite tube test chamber is shown in Figure 3.

The minimum explosive concentration or the lower explosive limit of a dust sample is also determined in the Hartmann Apparatus, except that an induction spark igniting source is used instead of the timed condenser discharge spark. A weighed amount of dust is spread in a thin layer in the dispersion cup. The top of the Hartmann tube (lucite) is covered with a filter paper diaphragm held in-place by a locking ring. A 0.16-cm hole is made in the center of the filter paper to prevent pressure build-up in the tube from the dispersing air and the tungsten electrodes are adjusted to a gap length of 0.48 cm. The electric spark is struck and the current adjusted to 23.5 milliamperes. The dust cloud is formed in the lucite tube by releasing air from the 1.31-liter reservoir through the full-port solenoid valve; optimum air pressure is 69 kPa.

Following ignition of the dust, sufficient pressure must develop to burst the filter paper diaphragm; appearance of flame in the tube is not considered propagation. The pressure required to burst the paper diaphragm is about 21 kPa, depending on the rate of pressure rise. If propagation occurs for a given weight of dust, the weight is reduced by a 5-milligram increment and another trial made until a quantity is obtained which fails to propagate the flame in any of four successful trials. The lowest weight at which the flame propagates is used in calculating the minimum concentration. Tests are made with the electrodes 10 cm from the bottom of the tube.

Figure 4 is a schematic of the Hartmann Apparatus for determining the minimum concentration for explosion of a dust cloud, and figure 4A shows the test setup.

Minimum Ignition Temperature of a Dust Cloud

The minimum ignition temperature of a dust cloud is determined in the Godbert-Greenwald Furnace. This furnace consists of a 3.7-cm diameter vertical alundum tube, 23 cm long, wound with 6.4 meters of 18-gauge nichrome V wire. The windings are placed closer together toward the two ends than in the middle of the tube to obtain relatively even temperature throughout. The tube is mounted between two 1.3-cm thick transite plates in a 15-cm diameter sheet metal cylinder with kieselguhr packing between the alundum tube and the sheet metal shell. A glass adapter connects the top of the tube to a small brass chamber that has a hinged lid for inserting the dust. A full port solenoid valve between the dust chamber and a 500-mm air reservoir controls the dispersion of the dust. The air reservoir is connected to a compressed air line and a pressure gauge. Dust contained in the brass chamber is dispersed downward through the furnace by a single blast of the compressed air from the reservoir. Dispersion pressure is 21 kPa. The weight of sample placed in the brass chamber is varied in accordance with the concentration desired in the furnace. This apparatus is depicted schematically and pictorially in Figures 5 and 6.

The temperature of the furnace is measured with a 22-gauge chromel-alumel thermocouple which is located 0.08 cm from the furnace wall at mid-height. An automatic temperature controller maintains the furnace temperature at the desired value. Ignition is denoted by the appearance of flame below the mouth of the furnace. The ignition temperature is the minimum furnace temperature at which flame appears at the bottom of the furnace in one or more trials in a group of four. Furnace

temperature is varied in 10°C increments; the highest temperature attained in the furnace is 750°C.

Part B Tests

This segment in Phase I of the program required that the Hartmann Apparatus be modified to allow for the control of air temperature and relative humidity. A complete description of this modification was presented earlier under the Test Air Environmental Control Methods section. Dust cloud concentrations were maintained at five times the minimum concentrations for explosion established in the Part A tests. Once again, the criteria for selecting the threshold minimum ignition energy level was four negative results below the last positive result for either a 0.05- or 0.80-joule energy interval.

The environmental test air conditions for the Hartmann Apparatus for this part of the tests were 24°C at 2, 50 and 76 percent relative humidities and 52°C at 2, 39 and 58 percent relative humidities.

Part C Tests

The unmodified Hartmann Apparatus was used for this part of the tests to determine the effect of dust concentration on minimum ignition energy. Dust concentrations of three and one times the minimum concentration for explosion were used. Test air was maintained at 24°C and one percent relative humidity. Previously described test procedures were followed.

Test Results

Tables 5 through 7 present the results of the tests performed in Part A of this phase of the program, while Tables 8 and 9 present results for Parts B and C, respectively. Figures 7 through 28 graphically present the data contained in all five tables. Tables 10, 11 and 12 summarize the findings of this phase of the program.

Part A Test Results

Minimum Explosive Concentration

Results of this test series are presented in Table 5 and Figures 7 and 8. It is seen that 200-mesh M-1 has the lowest minimum concentration for explosion followed by 200-mesh M-30, 100-mesh Composition B and Classes 1 and 3 HMX. Figure 7 shows that M-1 and M-30 had their highest minimum concentrations for

explosion at the smallest particle sizes. This phenomenon is explained by the observed tendency for the 44-micron particles to hold an electrostatic charge and agglomerate. The net effect is that they cling to themselves and behave as much larger particles in the test apparatus.

Composition B appears to behave differently from the two propellants. Its minimum concentration for explosion decreases as its particle size increases. This is also attributed to the electrostatic charging phenomenon. HMX reaches its lowest value of minimum concentration for explosion at Classes 1 and 3 particle sizes.

Minimum Ignition Energy of a Dust Cloud

Table 6 presents the results of this series of tests. It is apparent that M-1 and Composition B are the two most sensitive materials followed by M-30 and HMX. Figures 9 and 10 show M-1, M-30 and Composition B plateau out at the 44- and 74-micron particle size ranges. As expected, the highest minimum ignition energies occur at the largest particle sizes for all four materials. The general rule that the minimum ignition energy increases with increasing sample particle size is followed in this case.

Minimum Ignition Temperature of a Dust Cloud

Results of the minimum ignition temperature tests for dispersed dust clouds of the four sample materials are presented in Table 7. Variations in ignition temperature as a function of particle size and dust cloud concentration are presented. Figures 11 and 12 graphically illustrate the effect of particle size on ignition temperature for a fixed dust cloud concentration. M-1 has the lowest minimum ignition temperature (210°C) followed by M-30 (230°C). Both of these minimums occurred at the 44-micron particle size range. Composition B ignites at 360°C and 44-micron particle size. Reducing the loading by 0.1 gm/l resulted in a 30°C drop in ignition temperature. Class 1 HMX ignited at 350°C at a 0.92 gm/l concentration. It is apparent from the broad ranges of ignition temperatures found in Figure 12 that the ignition temperature of HMX is very sensitive to particle size and dust concentration. The spread of ignition temperatures for HMX lies between 350°C and 750°C.

Part B Test Results

Minimum Ignition Energy, Controlled Environmental Air

Table 8 presents the results of the experimental effort performed on this part of Phase I of the program. Figures 13 through 20 present minimum ignition energy as a function of relative humidity for the four samples evaluated. Figures 21 through 24 show the effect of particle size on minimum ignition energy. Careful study of Figures 13 and 14 reveals that the trend is for the minimum ignition energy of M-1 propellant to increase with increasing relative humidity. The lowest value of minimum ignition energy for M-1 always occurs at the 2 percent relative humidity point. Variations in ignition energy are significant between humidity levels at fixed temperatures.

Figures 15 and 16 present the results of M-30 propellant. The same trend is seen to exist; increased ignition energy at increased relative humidity. A minimum value of ignition energy occurs at the 44-micron particle size range at the 2 percent relative humidity level. Comparison of M-1 and M-30 data reveals that the M-1 is more sensitive (0.20 joule) than the M-30 (0.30 joule) at the 2 percent relative humidity values.

Composition B results, which are plotted in Figures 17 and 18, follow the same pattern established by the two propellants. The lowest value of ignition energy (0.20 joule) occurs at the finest particle size, 44 microns, and the 2 percent relative humidity value. Relative humidity and temperature affect the results by factors of up to 20 times the minimum value of 0.20 joule.

The results of the minimum ignition energy tests on HMX are presented in Figures 19 and 20. Values of ignition energy greater than 8 joules have been plotted as 8 joules to simplify presentation of the data. A general trend exists for the ignition energy; i.e., to increase with increasing relative humidity. The lowest value of 1.60 joules occurs at the 2 percent relative humidity value for the Class 1 material. Throughout this section of the report, it may be noted that the HMX data is the least consistent of the four materials tested. This is attributed to the broad range of particle sizes found in the Classes 1 and 3 material (see Table 2). The reproducibility of each data point is dependent upon the particle size distribution of each test sample. The broader this distribution, the more random will be the results of the ignition energy tests.

Figure 21 presents plots of minimum ignition energy vs. particle size for M-1 propellant at two temperatures and three relative humidities. It is seen that for the 52°C data, the lowest ignition energies for a given relative humidity curve occur at the 74-micron particle size range. Differences between the 39 percent and the 2 percent relative humidity values disappear at the 149-micron particle size range. Examination of the 24°C graph reveals that the highest ignition energy values occurred at the 74-micron particle size point. This result is contrary to all of the other data observed in this part of the tests. The reason for this discrepancy is not known.

Results of the tests on M-30 are presented in Figure 22. The trend is for the minimum ignition energy to increase with increasing particle size. Definite differences in results due to relative humidity are readily apparent. In general, increasing the relative humidity and particle size increases the minimum ignition survey.

Composition B test results are shown in Figure 23. Particle size has little effect in the 44- to 74-micron range. Above these values, the ignition energy increases substantially. Looking at the 24°C graph, it is seen that the minimum energy increases from 0.20 joule at 2 percent relative humidity to 1.60 joules at 76 percent relative humidity. This is a significant factor because, in a process operation, it is likely that an ungrounded conductive element can store and discharge 0.20 joule. However, it is highly unlikely that the same piece of equipment will be able to accumulate 1.60 joules of electrical energy.

Figure 24 shows the variation in minimum ignition energy with particle size for HMX at two air temperatures and three relative humidities. The lowest values of ignition energy occur at the Class 1 points. It is seen that the most sensitive condition (1.60 joules) occurs at the Class 1 size at 2 percent relative humidity and 24°C air temperature.

Part C Test Results

Minimum Ignition Energy at Reduced Concentrations

Table 9 presents the results of the test effort expended on this part of Phase I of the program. Figures 25 through 28 graphically illustrate the data contained in the table. All of the data in these four figures have the same characteristic; namely, the minimum ignition energy increases markedly as the concentration is decreased from 5 to 3 to 1 times the minimum concentration for explosion. For example, M-1 at

149-micron particle size has a minimum ignition energy of 0.25 joule at 0.25 gm/l. This value rises to 1.60 joules at 0.15 gm/l and is greater than 8 joules at 0.05 gm/l (the minimum concentration for explosion). The significance of this finding is readily apparent in hazards analysis work. It is currently common practice to evaluate dust explosion hazards by calculating the electrical energy available in a given process operation and comparing this value with the minimum ignition energy of the material. No allowance is made for the fact that the minimum ignition energy is based on concentrations that are 5 to 10 times the minimum concentration for explosion. This oversight introduces factors of safety which are so large that they make the calculation of the probability of the event occurring a fruitless exercise. Data collected in Phase I of the program should be useful to the hazards analysis investigators because it will allow them to more realistically evaluate specific situations.

Conclusions

As a result of the dust phase explosion tests performed on M-1, M-30, Composition B and HMX, it is possible to conclude the following:

1. M-1 has the lowest minimum concentration for explosion followed by M-30, Composition B and HMX.
2. Particles of M-1 and M-30 below the 74-micron range tend to agglomerate and cling to themselves due to an electrostatic charging phenomenon. The result is that they yield higher minimum concentrations for explosion than the 74-micron particles.
3. The minimum ignition energy increases with increasing sample particle size. M-1 and Composition B are the two most sensitive to electrostatic discharge followed by M-30 and HMX.
4. As the particle size increases, the minimum ignition temperature increases. M-1 has the lowest minimum ignition temperature followed by M-30, Composition B and HMX.
5. The minimum ignition energy of all four materials increases with increasing test chamber relative humidity. Lowest values of ignition energy occurred at the 2 percent relative humidity point for each of the four materials tested.

6. The minimum ignition energy of the four materials increases markedly as the dust cloud concentration is decreased from 5 to 3 to 1 times the minimum concentration for explosion.
7. Hazards analysis of electrostatic discharge potentials in a given process must be based upon consideration of the effect of concentration on minimum ignition energy (current practice is to use ignition energies which are obtained at 5 to 10 times the minimum concentration for explosion).

Recommendations

It is recommended that implementation of the following items be considered:

1. Results obtained from this phase of the program can be incorporated into a standard hazards analysis data bank.
2. Additional explosive and propellant materials should be tested for the effects of particle size, dust cloud concentration and relative humidity on the minimum concentration for explosion, minimum ignition energy and minimum ignition temperature.
3. Obtain dust samples from in-process operations at Army Ammunition Plants and establish the potential explosion hazard by comparing the sampled concentrations with the measured data generated by this program.

PHASE II - EXPLORATORY STUDY OF M-1 PROPELLANT DUST EXPLOSIBILITY

Equipment and Test Procedures

The purpose of this phase of the program was to conduct a preliminary investigation into the effects of certain propellant dust parameters on minimum explosive concentration and minimum ignition energy: dust particle size, air temperature, moisture content, and introduction of solvent into the propellant dust. This section of the report will describe the equipment and experimental techniques used in the performance of the test evaluation of M-1 propellant dust explosibility. It should be noted here that procedures used by the Bureau of Mines were studied and used when feasible.

M-1 Propellant Dust Production

It was noted earlier that a parallel investigation being conducted by ARRADCOM was concerned with collecting typical dust samples at the operation ammunition plants. Since these dust collections were not yet available, it was deemed necessary for the experimental program to grind M-1 propellant grains to the suitable particle sizes for explosibility investigations. For the tests to be conducted in Phase II, the dust was obtained from single perforated M-1 propellant grains.

A grain of propellant measures 0.1143 cm in diameter by 0.49 cm long and has a single 0.032-cm diameter hole in the center. The bulk density is approximately 625 kg/m³. M-1 contains 84.2 percent nitrocellulose (13.15% N), 9.9 percent dinitrotoluene, 4.9 percent dibutylthalate and 1 percent diphenylamine.

In the manufacture of M-1 propellant, large amounts of M-1 dust particles are produced during the drying process, possibly in explosive concentrations. Compounding the problem is the presence of ethyl alcohol and ether vapors evaporating from the M-1 propellant, and the dust particles in the propellant plant can vary greatly in moisture, solvent content and size. Therefore, to exactly simulate the dust environment in a propellant plant would be most difficult; hence, the test procedures had to be designed in a way that the contribution of each parameter to dust explosibility can be discerned. The information gained about each parameter can then be combined to assess the explosibility situation in the total propellant plant environment.

The production of M-1 dust in the laboratory was made difficult by two properties of M-1. First, M-1 is highly flammable and could possibly be ignited by sparks or excessive shock. Most grinding procedures utilize shock or friction to produce dust. This meant that a trade-off between grinding efficiency and possibility of ignition had to be made. Second, the M-1 pellets would tend to split along the longitudinal axis; thus, the long fibers generated were too flexible to grind into small particles. Initially, attempts were made to grind the propellant using a mortar and pestle. After failing with mortar and pestle, a Wiley ball mill tumbler was used to grind the propellant. Needed for the use of the ball mill is water and ice which act as a lubricant and also serve to extinguish possible fires. After three days of tumbling, the M-1 had been ground to a point where approximately 1/3 to 1/5 of the propellant load would pass through a Tyler No. 150 mesh sieve; i.e., 2/3 or 4/5 of the dust particles were larger in size than 105 microns (1.04×10^{-2} cm). Succeeding attempts to grind M-1 propellant in the ball mill were unsuccessful in duplicating the dust size distribution; hence, the procedure was abandoned.

The next grinding attempt utilized a paint shaker. Two capsules containing approximately 50 grains each of M-1 and water, and suitably sized ceramic balls were agitated on a paint shaker for approximately 20 minutes. The resultant wet propellant particles were dried under a heat lamp and sieved to obtain size-to-sample mass distribution which is shown in Table 13. Also, the sieve mesh sizes are the same as those used in Phase I and are listed in Table 3.

It was pointed out in the "Discussion of Dust Explosion" suction that particle sizes between 1 and 120 microns closely followed Stokes Law and that these would be the most likely dust particle sizes to be found in an ammunition plant drying operation. Therefore, it was decided that the tests in Phase II would be conducted using two particle sizes from the grinding operation: dust caught between No. 140 and No. 200 sieves (dust particles between 75 and 105 microns) and dust passing through the No. 200 sieve (75 microns).

Two dust moisture contents were also tested. These two dust moisture types were termed "wet" and "dry". Wet dust is dust that had not been placed in a desiccator with an average moisture content by weight of about 3 percent. Dry dust is dust that is placed in a desiccator with a moisture level approaching 0 percent.

To produce dust particles that contained solvent (either ethyl alcohol or anhydrous ether), a quantity of solvent was added to a weighed quantity of dry propellant dust. This "solvent wet" propellant dust was dried in airtight containers prior to testing. Immediately before testing, the solvent wet propellant was sieved to break up the adhering particles, allowing more solvent to escape, lowering the solvent content below 10 percent by an unknown amount.

Ignition Source

Since the Bureau of Mines (Ref 40) has a standardized test program and equipment for dust explosibility tests, Southwest Research Institute implemented these standards into the test program. A Hartmann Apparatus was used for the explosibility tests. The ignition source was an electrical discharge spark, produced by an apparatus consisting of a 100/400-v power supply, luminous tube transformer and a capacitor bank. The capacitance of the capacitor bank could be varied in 1-microfarad increments, allowing the stored electrical energy to be varied. The voltage applied to the capacitors was either 100 or 400 volts, depending on the energy level desired. The capacitors, upon being charged by a power supply, would be disconnected from the power supply and discharged into the luminous tube transformer. The high voltage output from the transformer is passed through an air gap in the test chamber where ignition of the dust occurs. The energy of the spark in the test chamber is calculated as $1/2 CV^2$ where C is the capacitance of the capacitor bank and V is voltage across the capacitor bank. It is realized that this value is somewhat high due to losses in the transformer and that some energy remains in the capacitor bank. Thus, the calculated value of energy is a relative, not absolute value (Ref 2). The use of the electrical discharge system required timing between production of the dust cloud and release of the electrical spark. To understand this delay, the Hartmann Apparatus needs to be described (Appendix A).

Flame Propagation Determination

All tests were conducted with dust samples through the No. 200 sieve (less than 75 microns) and with a moisture content less than 5 percent. A dust sample was placed in a desiccator to achieve the low moisture level.

The dispersed dust, upon ignition, would burn with varying intensities, ranging from no visible reaction to detonation with rupture of the paper diaphragm. An immediate problem visualized was: what should be used as criteria for propagation of flame in

the dust cloud? It was decided that the same propagation criteria would be used as those of Reference 3. A summary of the most important points stated by the Bureau of Mines Report 5624 in the use of the Hartmann Apparatus for determination of minimum explosive concentration and minimum ignition energy is as follows:

A. Determination of Minimum Explosive Concentration

1. Mass of dust placed in Hartmann Apparatus should be varied in 5-milligram increments.
2. The spark gap should be 0.5 cm. Tests should be made with the electrodes placed 5 cm and then 10.2 cm from the bottom of the lucite tube.
3. The spark should be a continuous induction spark of 23.5 milliamperes.
4. The reservoir air pressure should be varied until an optimum value is found.
5. Propagation of flame occurred only if the paper diaphragm had ruptured.

If the diaphragm ruptures, the dust amount is decreased by 5 mg and the testing continues until there is no diaphragm rupture in four successive trials for a given weight of dust. The lowest weight at which the diaphragm bursts is used to calculate the minimum explosive concentration. Tests should be made with the electrodes 5 cm and then 10.2 cm from the bottom of the tube. The average of the two weights thus obtained (one at 5.08 cm and one at 10.2 cm) is divided by the volume of the reaction chamber for the value of the minimum concentration.

B. Determination of Minimum Ignition Energy

1. Mass of dust placed in the Hartmann Apparatus should be kept constant at a level corresponding to 5 to 10 times the minimum explosive concentration.
2. The spark gap width should be varied to find an optimum value.

3. The spark should be obtained from a single capacitative discharge. The capacitors discharge through a luminous tube transformer.
4. The reservoir air pressure should be varied until an optimum value is found.
5. A 10.2-cm flame is considered propagation of flame. Tests are repeated at a given energy level until propagation occurs or four tests have been conducted. If propagation occurs, the ignition energy is lowered by decreasing the capacitance by 1 microfarad (if energy is less than 50 millijoules, and four if above) and up to four tests are tried at this new ignition energy. This process is repeated until no propagation occurs in four trials. The lowest energy at which propagation occurs is the minimum ignition energy.

It should be noted that only a 10.2-cm flame is considered propagation for minimum ignition energy tests and a ruptured diaphragm is required for propagation in the minimum explosive concentration tests. Also note how factors inherent to the test apparatus itself (spark gap, suspension pressure, delay time) are made constant by choosing optimized values; i.e., values which would most likely encourage ignition. Again it should be realized that the values obtained with the Hartmann Apparatus may be different from those obtained in a real life situation. However, the Hartmann Apparatus will accurately determine the explosibility of one dust sample relative to another (Ref 40). It can be argued that visual determination of propagation is not the best method; therefore, the possibility of using pressure-time traces as a supplement to the propagation criteria is considered in Appendix B.

Photographic Coverage of Tests

Representative tests were photographed with a high speed camera at 64 frames per second. Both tests in which propagation occurred and tests in which no reactions occurred were photographed. The films show clearly that the dust samples were fully dispersed and relatively uniform at the moment ignition was attempted (see Figures 29 through 33).

Establishing a Test Matrix

In the manufacture of M-1 propellant, a large amount of propellant dust is produced. An especially critical operation is

the drying of freshly extruded M-1. Wet M-1 contains water, ethyl alcohol and anhydrous ether, the latter two extremely flammable. The drying process creates propellant dust with varying amounts of solvents in the dust particles and disperses solvent vapors in the air, potentially a hazardous situation. In an addendum to an existing contract, Southwest Research Institute was to determine the minimum ignition energy in the form of an electrostatic discharge for M-1 dust in relation to five parameters, each at three different values: dust concentration, moisture content, air temperature, particle size and solvent content. It was seen that a test matrix of 3^5 or 243 test parameter combinations were possible without considering spark energy variations. The number of combinations could be reduced by the use of fractional factorial design. The advantages of a fractional design are that the important parameters and their combinations are identified without the need for a time consuming probe of the full matrix, and testing of unimportant parameter combinations is minimized. In this fashion, it is possible to isolate sub-regions of the full matrix where strong functional relationships exist, which can be explored in more detail if warranted. Functional factorial design is based on statistics and a discussion of the technique is contained in Davies' book (Ref 41).

In the initial experimental design, the full matrix 3^5 parameter combinations were reduced by a factor of 1/3 to 81 combinations. At this level, all main effects and all two-factor interactions are clear of one another; i.e., they are not confounded. Higher order interactions are assumed to be negligible. All of the parameter combinations in this 1/3 fractional matrix are shown in Table 14 where the superscripts 0, 1 and 2 represent low, medium and high levels of concentrations (C), temperature (T), particle size (P), moisture (M) and solvent (S). The three values initially decided upon for each parameter are:

C (dust particle concentrations): 50, 250 and 500 mg/l

T (air temperatures): 24°C, 38°C and 52°C

P (particle sizes): particles ≤ 75 , ≤ 105 and ≤ 210 microns

M (moisture content by weight): 0%, 5% and 10%

S (solvent content by weight): 0%, 10% ethyl alcohol and 10% anhydrous ether.

It was decided that each of the 81 parameter combinations would be tested at three different energy values: 0.5, 2.0 and 8.0 joules. This made a total of 81 times 3 or 243 tests. It was realized at this time that the test program above was too large to be carried out within the imposed limits of time and funds. Further reduction of the test program was necessary.

The test matrix was revised after studying literature on Bureau of Mines procedures in dust explosibility testing (Refs. 40 and 42). It is helpful to examine the Bureau of Mines' explosibility testing procedure. For this section of the program, dust explosibility will be defined as the ease with which a dust cloud can be caused to produce an explosion. Dust explosibility is difficult to define quantitatively because of its dependence on many parameters and because explosion criteria are themselves inexact. To have meaning, there must be a way to relate the explosibility between different dusts (say, for example, flour and coal dust). The Bureau of Mines has developed a quantitative expression for dust explosibility, the Index of Explosibility, in which the explosibility of a certain dust sample is compared to a standard dust sample; i.e., Pittsburgh coal dust. An analogy is found in relating the explosive power of various explosives to that of TNT (TNT Equivalency Tests). All explosibility tests are conducted with dust through the No. 200 sieve (particles \leq 75 microns) and with a moisture content less than 5 percent. The Index of Explosibility is computed as shown below:

$$\text{Index of Explosibility} = \frac{\text{Ignition Sensitivity} \times \text{Explosion Severity}}{\text{Ignition Temperature} \times \text{Minimum Ignition Energy} \times \text{Minimum Explosive Concentration (Pittsburgh Coal Dust)}}$$

where

$$\text{Ignition Sensitivity} = \frac{\text{Ignition Temperature} \times \text{Minimum Ignition Energy} \times \text{Minimum Explosive Concentration (Pittsburgh Coal Dust)}}{\text{Ignition Temperature} \times \text{Minimum Ignition Energy} \times \text{Minimum Explosive Concentration (Dust Sample)}}$$

and

$$\text{Explosive Severity} = \frac{\text{Maximum Explosive Pressure} \times \text{Maximum Rate of Pressure Rise (Dust Sample)}}{\text{Maximum Explosive Pressure} \times \text{Maximum Rate of Pressure Rise (Pittsburgh Coal Dust)}}$$

For all three dimensionless quantities defined above, a value greater than 1 indicates increased danger over coal dust; while a value less than 1 indicates a decreased danger relative to coal dust. All three quantities are calculated from specific test results: minimum ignition energy, minimum ignition temperature, minimum explosive concentration and explosion pressure-time traces.

The result of the Bureau of Mines' procedure is information relating dust samples having standardized parameters of particle size and humidity through comparison with Pittsburgh coal dust. Furthermore, these dust parameters and procedures of testing are chosen such that the explosion probability is maximized and functional dependence on the parameters is constant in the region of the values of the parameters.

The test program initially to be conducted by Southwest Research Institute can be seen to be only a portion of the Bureau of Mines' dust explosibility testing process. It was seen that a compromise had to be made between obtaining the functional relationship between the dust parameters in relation to explosibility over a range of values (originally three values) and the probing over a very small range with a large number of repetitive tests to comply with the Bureau of Mines' procedures for determination of minimum ignition energy and explosive concentrations. Furthermore, it was seen that for a propellant plant, the most useful test data would be minimum ignition energy and explosive concentration as found by Bureau of Mines' procedures in relation to the parameters of particle size, and solvent and moisture contents.

A test program was chosen which represented a three-way compromise between the original Scope of Work, procedures used by the Bureau of Mines and limitations of time, money, dust supply and equipment availability. For the test program, it was decided that the test matrix would contain the effects of three parameters in relation to minimum explosive concentration and ignition energy. The three parameters are propellant dust particle size, moisture content of the dust and the addition of solvent to the propellant dust. Two dust particle sizes were used: particles 75 microns (through No. 200 mesh sieve) and particles in the size range of 75-105 microns (dust particles caught between the No. 140 mesh and the No. 200 mesh sieves). Two moisture contents were used: 0 percent moisture content by weight (dust was placed in a desiccator) and approximately a 3 percent moisture content by weight (dust not placed in a desiccator). Three solvent variations were used: the addition

of no solvent, addition of 10 percent ethyl alcohol by weight and the addition of 10 percent anhydrous ether by weight.

The parameters listed above can be combined into a $3 \times 2 \times 2$ test matrix. Only 7 of the 12 test cases were used in the test series. This is without considering ignition energy or dust concentration values.

The test series conducted differed from the Bureau of Mines' procedure in four major aspects. For determining minimum explosive concentration, a single capacitative discharge was passed through a luminous tube transformer rather than through a continuous induction spark. It can be argued that the use of a single-timed spark can be as effective as a continuous discharge, provided that a proper delay time is used and the spark has sufficient energy. Also, minimum explosive concentration tests were conducted using electrodes placed 10.2 cm above the bottom of the lucite tube and not at both, a 10.2-cm and then a 5.08-cm height as specified. A third difference is the use of 5-milligram increments in determining minimum explosive concentration and 1- to 4-microfarad increments to vary energy for minimum ignition energy determination. Finally, the four trial verification procedures were not followed. The reason for the last three differences is the large number of tests which would have been required.

To determine minimum explosive concentration and minimum ignition energy for a one-test case would require about 100 tests or 1,200 tests for the whole program, if the Bureau of Mines' procedure is followed exactly. Realizing that this number of tests could not be conducted within the imposed limits of time, budget or propellant dust supply, only selected tests were fired from each test case. For purposes of an exploratory set of tests, this procedure would give some estimate of minimum explosive concentration and minimum ignition energy.

Test Results

To open our discussion of the test results and to better understand the operation of the Hartmann Apparatus, reference is made to Figures 29 through 33. Here, through a series of high speed motion picture frames, the dispersion of the dust cloud in the test chamber can be clearly seen. From selected frames, an attempt is made to illustrate first, the early state of M-1 dust rising in the test chamber (Fig. 29), then, the rising of dispersion of the cloud (Figs. 30, 31 and 32), and finally, the occurrence of an explosion wherein the paper diaphragm at the top of the test chamber is ruptured (Fig. 33).

In Appendix C, the detailed data for all 146 tests are tabulated. In the table, the following are listed: date, test number, mass of propellant (mg) placed in the Hartmann Apparatus, size of screen through which particles are sieved, moisture or solvent content in percent by weight, the type of solvent used, the air temperature in degrees centigrade, the delay time between release of compressed air and spark in milliseconds, the spark gap in centimeters, the suspension pressure in pascals, capacitance of capacitor bank in microfarads, the voltage across the capacitor bank in volts, the stored energy in the capacitor bank (equal to $1/2 CV^2$) and the test results. For purposes of our discussion, the significant data of the test program for the seven test cases are broken down in Tables 15 through 21. In the tables are listed: test number, the concentration of the dispersed dust in milligrams per liter, the ignition energy in joules and the test results. Also, the tables divide the data into two categories:

1. Data concerning minimum ignition energy, and
2. Data concerning minimum explosive concentrations.

It may be noted that some tests appear in both categories; their values of energy and dust concentration make this possible.

Referring to Table 15, it can be seen that the minimum ignition energy for dry M-1 dust ≤ 75 microns ($\leq 7.62 \times 10^{-3}$ cm) in size is ≤ 0.15 joule. The minimum explosive concentration lies between 203 and 244 mg/l. Referring to Table 16, the minimum ignition energy for dry M-1 dust 75-105 microns (7.62×10^{-3} to 10.2×10^{-3} cm) in size is ≤ 0.175 (≤ 0.13 ft-lb) joule. The minimum explosive concentration lies between 122 and 244 mg/l.

For Test Cases III-IV shown in Tables 17 through 21, the highest dust concentration tested was 366 mg/l. Table 17 lists the data for wet M-1 propellant dust, 75-105 microns in size and at 3 percent moisture content. The minimum explosive concentration is apparently above 366 mg/l and there are insufficient data for determination of minimum ignition energy.

Tables 18 through 21 involve dust with solvent introduced. The data from these tables can be discussed together because the results are the same for each test case.

No propagation occurred in any of the tests used in the last four test cases. It was expected that the addition of solvent to the dust would lower the minimum ignition energy. This, however,

was not observed (compare Tables 15, 16 and 17 with Tables 18 through 21).

In using propagation criteria, it was found from tests that usually the dust cloud either reacted in the most severe manner (burst the diaphragm with an accompanying "bang") or had a minimal reaction (small localized flame around the spark gap). There were few tests where propagation criteria were satisfied without diaphragm rupture. There are several questions; i.e., problems that arose during the course of the testing program which should be mentioned, as they may have already occurred to the reader. One involves the question of applicability of the test results to real life situations. It is believed, however, that the optimum conditions created in the Hartmann Apparatus reflect the most dangerous situation possible for a given dust sample. A problem was encountered in the repeatability of tests. It was found that tests could not always be repeated from day to day. Also, there is the question of the effects of the addition and subsequent evaporation of water or solvent on M-1 propellant. The effect on explosibility is unknown. Finally, there is the question on the true particle size of the dust cloud in the Hartmann Apparatus. The dust particle sizes are known because they pass through a given sieve size. But the particles are then allowed to come in contact and adhere to each other. A certain amount of energy is needed to break apart these adhering particles and it is doubtful that it is fully accomplished in the Hartmann Apparatus. The method used in the tests was to sieve the dust immediately before the test in hope of breaking apart the adhering particles.

Both K.N. Palmer (Ref 39) and the Bureau of Mines (Ref 40) address themselves to these questions. Nevertheless, the Bureau of Mines' procedure is the most used and respected technique, and with the recognition of the above mentioned problems, can provide some important data to be used by those in dust explosion prevention work. In Figures 34 and 35 are shown the explosibility curves for dry M-1 dust (<75 microns and also 75-105 microns in size). For both of these figures, ignition energy is plotted as a function of dust concentration. In Figure 34, a large number of data points are plotted to show the transition between no reaction and an explosion. The data points termed "localized reaction" and "burning" are cases of minimal or threshold reaction, and would be termed "no reaction", using Bureau of Mines' criteria. In Figure 35, the Bureau of Mines' criteria are strictly adhered to; hence, the apparent "sharp" transition between no reaction and explosion.

Conclusions

As a result of the tests performed in the M-1, M-30, Composition B and HMX Program, it is possible to conclude the following:

1. Explosibility of M-1 dust appears to increase with a decrease in particle size.
2. Explosibility of M-1 dust appears to decrease with addition of moisture to dust.
3. Particles \leq 75 microns in size have a minimum ignition energy of approximately 0.15 joule, and the minimum explosive concentration lies between 203 and 244 milligrams per liter.
4. Larger M-1 dust particles between 75 and 105 microns have a minimum ignition energy of approximately 0.175 joule, and the minimum explosive concentration lies between 122 and 244 milligrams per liter.
5. These values are more than an order of magnitude lower concentration than the explosive concentrations of the non-oxidizing coal dust at 2,000 mg/l.
6. A pressure trace can be used on the Hartmann Apparatus as an aid in determining propagation. Also, its value would be greatest in the tests in which nominal propagation occurs, which is infrequent.

Recommendations

The exploratory study of M-1 propellant dust explosibility was most definitive in that it demonstrated that severe flash fires and explosions can indeed be initiated under certain sets of ambient conditions. Because of the exploratory nature of this study, it was possible only to identify the pertinent parameters which contribute to making an M-1 dust cloud susceptible to an explosive reaction. It was not possible to explore, in detail, each of the quantitative values of these contributing parameters; however, it was possible to arrive at specific recommendations for future investigations as follows:

1. Dust samples collected from the actual operations of the Army Ammunition Plants should be used in the experiments. There is reason to believe that

laboratory ground M-1 propellant does not produce representative dust specimens for true evaluation.

2. Some means should be developed for the introduction of solvent vapors into the test chamber in such quantities that they will represent the ambient environment at select points in the production plant.

PHASE III - EXPLOSIBILITY OF EXPLOSIVE AND PROPELLANT DUSTS

Equipment and Test Procedures

Dust Ignition Experiment Apparatus

An apparatus was constructed that allowed passing a spark discharge through a flowing dust-air mixture of uniform dust concentration (Figs 36 and 37). Dust concentration could be controlled and the procedure was to fix the spark energy at a sufficiently high value and vary the dust concentration to determine that concentration below which no ignition would occur. Minimum spark energies for HMX/TNT and RDX/TNT dusts have been measured by Willim and Nicholson (Ref 35), and have values of 0.016 and 0.222 joule, respectively. In addition, the study in Phase II by Gehring and Friesenhahn (Ref 2) on the explosibility of M-1 propellant dust indicates the steep region of the ignition energy vs. the dust concentration curve begins at about 0.3 joule. Thus, a spark energy 10 times this value (3 joules) was chosen in this study to insure operation on the steep part of the curve.

The apparatus was set up in a test chamber with control panels (Fig 38), oscilloscope and streak camera located in an adjoining room. Primary airflow through the dust fluidizer and secondary airflow used to dilute the primary dust stream to desired concentrations were registered on the control panel. The resulting dust-air mixture entered the vertical tube test volume from the top. A Tektronics Type 555 dual-trace oscilloscope was used to record output from photodiodes that monitored flame propagation. A Beckman and Whitley Model 370A streak camera also monitored flame motion. Valve openings and spark discharge were synchronized with a Jenor Model 2410 automatic programmer.

In the electrical circuit for the spark ignitor, the voltage was variable from 1 to 10 kilovolts and the capacitance was variable from 1 to 5 μ f in 1- μ f increments.

Dust Ignition Experiment Operation

Samples were taken from production grade RDX, HMX, M-1 propellant and Ball Powder. These materials were ball milled and particles larger than 250 μ m removed by dry sieving the M-1 and Ball Powder, and wet sieving the RDX and HMX with freon (RDX and HMX are not soluble in freon). Sieving was accomplished using either a sonic sifter (Fisher-Scientific) or large standard screens and brush. Residual moisture or solvent was removed from

the RDX and HMX by placing them in a drying oven at 80°C for one hour. These materials are not affected by 80°C temperature. Table 22 gives the composition of M-1 and Ball Powder used in the tests.

A dust sample was placed in the dust reservoir and the programmer energized to permit flow of air through the dust bed and into the 6-foot long, 2-inch I.D. plastic vertical tube (see Figs 36 and 37). Another signal started the firing sequence causing the programmer to automatically close control valves and, within 50 msec, discharge the capacitors through electrodes mounted in the column. Amount of energy in the discharge was calculated in the usual way using the $1/2 CV^2$ formula, although it is recognized that this value does not represent the true energy absorbed by the dust.

Dust concentration during a run was measured by collecting the dust exiting the vertical tube for a fixed time interval and relating this to the amount of airflow during this interval. Airflow was measured by rotameters in both the primary and secondary air lines. Dust concentration in this airflow was determined through measurements to stay within 10 percent of the average value throughout a test run. Variations in dust concentration between runs with the same initial conditions are within 20 percent.

Any propagating reaction in the dust column was detected both by the photodiodes and the streak camera. The time for a flame to pass between the two photodiodes was recorded so that the flame velocity could be calculated. A separate calculation of the flame velocity was made from the simultaneous streak photograph.

Manufacturing Site Sampling Apparatus

Air pumps were used to draw dusty air through millipore polyvinyl chloride (PVC) or cellulose ester filters that had been tared and placed in millipore aerosol analysis monitor holders. The flow rate through the filters was calibrated to a rotameter in the line to the pump. Calibration was accomplished by connecting a graduated 1,000-ml tube to the intake of the filters. The pump was operated for a long enough time so that the flow rate was stabilized. Then, a dish containing a soap solution was momentarily brought into contact with the bottom of the inverted intake and the time for the bubble-air interface to sweep out a particular volume of the graduated tube was measured (see Fig 39). This was converted to a liters-per-minute flow rate for that particular rotameter setting. In this way, a chart

relating rotameter setting to flow rate was constructed. All pumps were individually standardized.

Manufacturing Site Sampling Operation

At the manufacturing site, likely locations for sampling were noted during a walk-through inspection. The schedules of the workers, and magnitude and frequency of the operation were noted. A schedule was then devised to allow the sampling pumps to be set up in a way to cause minimum interference with production and to allow a sampling time of typically two hours or more. Most of the filters used were the polyvinyl chloride type as they are not as sensitive to humidity variation as are cellulose ester filters. After the pumps were emplaced and attendant filters connected, the pumps were started and adjusted to a flow between 1.5-2.5 liters/minute. The sample volume is the flow rate times the total pump operation time.

The used filters were weighed before leaving the installation. Unused filters, serving as controls, were also weighed so that balance differences and humidity effects could be accounted for. The quantity of dust collected divided by the sample volume provides the dust concentration.

Test Results

Results of Spark Ignition Studies

Table 23 summarizes the results obtained here with HMX and shows that for the 3-joule discharge, the minimum explosive dust concentration is 0.3gm/liter for particles in the 125-177 μm range and between 0.15-0.3gm/liter for particles in the 88-125 μm range. No ignition was observed for concentrations below 0.15 gm/liter or with larger particles. These results are in general agreement with the results obtained in Phase I by Hazard Research, Inc., with a Hartmann Apparatus(1), although exact agreement is not expected since the Hartmann Apparatus produces a relatively non-homogeneous dust cloud. They found for Class 1 HMX particles of median diameter 100 μm ranging from 65 to 320 μm the minimum explosive concentration was 0.5gm/liter. Considering their less homogeneous dust concentrations and their larger range of particle sizes, this result seems consistent with that obtained here.

Table 24 shows the results obtained with RDX. The minimum explosive dust concentration is 0.3gm/liter for particles in the range of 63-68 μm . For particles in the 177-250 μm range, ignition was not observed in the concentration ranges studied.

The RDX powder had a tendency to form clumps which effectively caused the particle size to change during an experimental run. Thus, the particle size associated with these results may not be the same as the initial particle size. However, the results are consistent with the HMX results where no agglomeration occurs.

Dusts that were roughly similar to those studied here were tested by Willim and Nicholson (Ref 35). They tested 60/40 RDX/TNT and 75/25 HMX/TNT mixtures by sparking dust clouds formed by passing a blast of air into a known amount of powder. No provision for observing flame propagation was made. For their RDX/TNT dust with particles ranging from 124-154 μm , they found a minimum ignitable dust concentration around 0.09-0.12 gm/liter. In contrast, the current results for RDX dust with particles ranging from 125-177 μm indicate a minimum explosive dust concentration of 0.75-1.5 gm/liter. The inconsistency in these results can be attributed to the lack of knowledge as to whether a flame would propagate through a low and relatively non-uniform dust concentration used in their experiment.

If the data obtained here is plotted as concentration vs. particle size (Figs 40 and 41), a region in which ignition can be expected becomes apparent. The average particle sizes for the ranges presented in Tables 23 and 24 were used in Figures 40 and 41. It is to be recalled that there is a general view that the ignitability becomes easier as the particle size decreases.

Table 2b gives results obtained with M-1 propellant. Minimum concentration for ignition is less than 0.06 gm/liter, but no subsequent flame propagation occurs through the low density dust. This value agrees with the value found in Phase I by Hazards Research, Inc. of 0.05-0.06 gm/liter using their Hartmann Apparatus (Ref 1). A minimum dust concentration of 0.3 gm/liter was sufficient for ignition followed by flame propagation in the particle size range of 44-150 μm . The minimum value lies between 0.15 and 0.3 gm/liter. This compares well with a minimum explosive dust concentration for the 75-105 μm particles of between 0.122 and 0.214 gm/liter found in Phase II by Gehring and Friesenhan of Southwest Research Institute (Ref 2). Severe agglomeration was encountered in testing Ball Powder. An ignition was observed for a concentration of 3 gm/liter, but no subsequent flame propagation occurred.

Although the minimum explosive dust concentrations are determined, there are factors that may occur in the manufacturing plants that can significantly change the minimum values. The most likely such factor is the presence of solvent vapor in the dust-air mixture. Solvent air mixtures of 0.05 gm/liter solvent

are detonable and the ignition energy for these gaseous mixtures is low. In addition, other modes of dust ignition are possible. An explosion from another source may generate a strong shock wave which propagates into the dust, causing ignition. This mechanism may be able to ignite concentrations or particle sizes that are not ignitable by a spark.

With regard to differentiation between deflagration and detonation during these tests, although all the ignitions reported were followed by flame propagation down the tube, no indication of deflagration to detonation transition was observed. The lucite vertical tube used as test volume was not destroyed during any of the propagations, indicating only low pressures were developed.

Streak photographs provide the best evidence that no transition to detonation occurred. An example of the appearance of a streak photograph taken of deflagration to detonation transition is shown in Figure 42. The mixture was stoichiometric propane-oxygen that was spark-ignited. Transition to detonation is indicated where the sharp change of slope occurs. Once started, the detonation propagates at great speed both into the partially reacted mixture already traversed by the flame front (called retonation) and into the undisturbed mixture. Representative streak photographs of flame propagation through three RDX powder dusts are shown in Figure 43. Flame propagation in the Bail Powder dust does not produce much light output, accounting for the very low light level of the photograph. All the streak records indicate flame speeds of approximately 300 m/sec, which is close to the velocity of sound in air. The flow field generated during an actual accident situation is very much more complicated than the flow induced in the vertical tube by the flame motion, so that transition in these dusts cannot be ruled out based on these results alone.

Results of Manufacturing Site Sampling Studies

Trips were made to Radford, Indiana and Milan Army Ammunition Plants, for the purpose of obtaining data on explosive dust concentrations which could be used, together with the laboratory ignition studies, to assess the hazards at these plants.

The operations investigated and the concentration of dusts observed at Radford Army Ammunition Plant are given in Table 26. These concentrations are significantly lower than that required for ignition. As has been shown, the concentrations of explosive

and propellant needed for ignition are about 0.15-0.3 gm/liter; therefore the values observed at Radford do not constitute an obvious hazard and some additional points must be noted. Most operations are batch-wide and are remotely controlled; hence, sampling must be controlled from a remote site or the sampling must be done over a time period greater than an actual batch period of operation. Thus, the results obtained may not be indicative of the maximum dust concentration that occurs during a batch run.

Data obtained at the Indiana Army Ammunition Plant from the sites indicated in Figure 45 are presented in Tables 27 and 28. Here again, the concentrations appear to be several orders of magnitude below that required for ignition; however, if, by some event, the dust reservoirs that accumulate near the bag-loading machinery were blown into the air, hazardous concentrations could be obtained. Based on conversations with maintenance personnel responsible for "blowing down" dust from weighing hoppers, there appears to be large lot-to-lot variations in dust levels, some lots being appreciably more dusty than those sampled here. It might be inferred from the frequency of "blowing down" the dust, that dusts must reach 4 to 5 times the levels determined during our sampling visit.

The data obtained at the Milner Army Ammunition Plant and presented in Table 29 also does not indicate a hazard. On the other hand, drilling and facing operations on M-105 shells generate a large quantity of dust which is exhausted through a 6-inch diameter aluminum collector tube to a dry scrubber. From conversations with the operator, it is estimated that 34 kg of dust are cleaned from 3 collectors per hour in the cyclone precipitators. Thus, the collecting line and precipitators contain enough dust that, if dispersed by some event, could easily achieve hazardous concentrations in air.

Conclusions

The following conclusions can be obtained from the results of the Phase III Program:

1. Studies of the ignition of energetic dust was conducted utilizing a flow system which permitted good control and characterization of dust concentration. The results are in essential agreement with other studies where only the overall dust concentration in a closed vessel, not the local concentration near the ignitor, was characterized (see Table 20). Dust concentration

required for ignition was roughly mapped out as a function of particle size.

2. Dust measurements made at Army manufacturing plants did not reveal any obvious dust hazards; however, additional observations indicate sufficient dust accumulates in collectors and around milling operations. A realistic assessment of potential explosion hazards from energetic dusts should consider the possibility that fires or explosions in the vicinity of such areas where dust has accumulated could disperse the dust into a cloud whose concentration is within the explosive range. Sources of fire or explosion could be regions of high solvent vapor concentration or the rupture of high pressure gas lines. The scenario for such an event would be the flow field from the fire or explosion lifts which fluidizes the dust particles lying on surfaces forming an explosive cloud which is ignited by the fire or some other source. Under these conditions, the ignition process could involve shock waves which may initiate detonation, rather than deflagration, in the dust cloud.

Recommendations

Based upon the results of the Phase III Program, the following recommendations are offered:

1. In collecting data at plant operations, longer duration of sampling is recommended to get a better idea of dust concentration.
2. Sampling of a pyrotechnic dust operation should be done to further study the hazard of fire and explosion.
3. Comparison of data obtained from a shock tube and the Hartmann Apparatus be extended for further verification of concluded results.
4. Data on ignition due to fire and shock are required to fully evaluate the explosion of accumulated dust which can stir up and explode by some external event.

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Table 1. Electrical spark energies and their significance^a

<u>Energy (joules)^b</u>	<u>Significance</u>
0.13 x 10 ⁻³	Minimum ignition energy for some flammable vapors
5 x 10 ⁻³	Minimum ignition energy for some dust clouds
7 x 10 ⁻³	Lead azide ignites
0.01	Minimum ignition energy for propellant dust layer (Netherland report 150 microns)
0.013	Minimum ignition energy for M-1 dust layer (Radford)
5-18 x 10 ⁻³	Energy in static electricity spark produced by human
0.15	Minimum ignition energy for M-1 dust cloud - Southwest Research Institute
0.25	Produces a heavy shock in humans
7.2	Threshold for producing ventricular fibrillation in humans
10.0	A human is in danger of death
11.03	Composition B ignites
12.5	Black Powder ignites
5 x 10 ⁸⁻¹⁰ 10	Lightning Bolt

^aReferences 5, 16, 17, 18, 19, 20

^bTo obtain energy in ft-lbs, multiply energy in joules by 0.74.

Table 2. HMX granulation specification

Through U.S. standard sieve No.	Class 1 %	Class 3 %	Class 5 %
12		99 min	
50	90 \pm 6	40 \pm 15	
100	50 \pm 10	20 \pm 10	
200	20 \pm 6	10 \pm 10	
325	8 \pm 5		98 min

Table 3. Listing of sieve and particle sizes

U.S. standard sieve series No.	Tyler screen scale equivalent mesh	Opening (micron)*
12	-	1,680
50	48	297
70	65	210
100	-	149
140	150	105
200	200	75
325	-	44

*1 micron = 9.906 x 10⁻⁵ cm.

Table 4. Volatile content of Composition B, HMX,
M-1 and M-30 samples

<u>Sample</u>	<u>Particle Size</u>	<u>% Volatiles</u>
Composition B	thru 100 mesh	0.27
	thru 200 mesh	0.53
	thru 325 mesh	0.40
HMX	Class 1	0.05
	Class 3	0.02
	Class 5	0.00
M-1	thru 100 mesh	0.56
	thru 200 mesh	0.51
	thru 325 mesh	1.30
M-30	thru 100 mesh	4.20
	thru 200 mesh	2.70
	thru 325 mesh	2.60

Table 5. Results of minimum concentration for explosion tests

<u>Sample</u>	<u>U.S. sieve number^a</u>	<u>Minimum explosive concentration (gm/l)</u>
M-1	100	0.05
	200	0.04
	325	0.06
M-30	100	0.07
	200	0.05
	325	0.12
Comp B	100	0.06
	200	0.08
	325	0.09
HMX	Class 1 ^b	0.50
	Class 3	0.50
	Class 5	1.00

^aMaterial passed through the sieve indicated.

^bTable 22 defines class particle size designations.

Table 6. Results of minimum ignition energy of dust cloud tests

Sample	U.S. sieve number ^a	Concentration (gm/l)	Min. ign. energy	
			Go (joule)	No Go (joule)
M-1	100	0.25	0.25	0.20
	200	0.20	0.20	0.15
		0.15	0.20	0.15
	325	0.30	0.20	0.15
		0.25	0.20	0.15
M-30	100	0.50	0.50	0.45
		0.35	0.40	0.35
	200	0.25	0.25	0.20
	325	0.60	0.20	0.20
Comp B	100	0.50	0.45	0.40
		0.30	1.60	0.80
		0.25	1.60	0.80
	200	0.40	0.20	0.15
		0.35	0.20	0.15
	325	0.45	0.20	0.15
		0.40	0.20	0.15
HMX	Class 1 ^b	2.00	0.45	0.40
	Class 3	2.00	5.60	4.80
	Class 5	2.00	4.00	3.20

^aMaterial passed through the sieve indicated.

^bTable 22 defines class particle size designations.

Table 7. Results of minimum ignition temperature of dust cloud tests

Sample	U.S. sieve number ^a	Concentration (gm/l)	Minimum ignition temperature (°C)
M-1	100	0.46	220
		0.37	230
	200	0.37	240
		0.27	240
	325	0.55	210
		0.46	210
M-30	100	0.92	250
		0.64	250
	200	0.92	230
		0.64	240
	325	0.92	230
		0.64	230
Comp. B	100	0.92	390
		0.55	330
		0.46	430
	200	0.92	360
		0.73	350
		0.64	380
	325	0.46	430
		0.92	360
		0.83	330
		0.73	360
HMX	Class 1 ^b	1.38	380
		0.92	350
	Class 3	1.38	690
		0.92	>750 ^c
	Class 5	1.38	580
		0.92	>750

^aMaterial passed through the sieve indicated.

^bTable 22 defines class particle size designations.

^cThe Godbert-Greenwald furnace could not be heated past 750°C.

Table 8. Results of minimum ignition energy tests under controlled environmental air conditions

Sample	U.S. sieve number*	Concentration (gm/l)	Temperature °C	Relative humidity (%)		Minimum ignition energy 60 (joule)	No Go (joule)
				52	58		
M-1	100	0.25	52	58	1.60	0.80	
		0.25	52	39	0.40	0.30	
		0.25	52	2	0.40	0.30	
		0.25	24	76	0.40	0.30	
		0.25	24	50	0.40	0.30	
		0.25	24	2	0.20	0.10	
		0.20	52	58	0.40	0.30	
		0.20	52	39	0.30	0.20	
200	0.20	0.20	52	2	0.20	0.10	
		0.20	24	76	0.80	C.50	
		0.20	24	50	0.80	0.50	
		0.20	24	2	0.30	0.20	
		0.20	52	58	0.50	0.40	
		0.20	52	39	0.40	0.30	
		0.20	24	76	0.50	0.40	
		0.20	24	50	0.40	0.30	
325	0.30	0.30	52	58	0.50	0.40	
		0.30	52	39	0.40	0.30	
		0.30	52	2	0.20	0.10	
		0.30	24	76	0.50	0.40	
		0.30	24	50	0.40	0.30	
		0.30	24	2	0.20	0.10	

*Material passed through the sieve indicated.

Table 8. Results of minimum ignition energy tests under controlled environmental air conditions
(cont'd.)

Sample	U.S. sieve number*	Concentration (gm/l)	Temperature °C	Relative humidity (%)	Minimum ignition energy No Go (joule)	
					50	60
M-30	100	0.35	52	58	3.20	2.40
		0.35	52	39	2.40	1.60
		0.35	52	2	1.60	0.80
		0.35	24	76	2.40	1.60
	24	0.35	24	50	1.60	0.80
		0.35	24	2	0.50	0.40
		0.25	52	58	1.60	0.80
		0.25	52	39	1.60	0.80
200	24	0.25	52	2	0.40	0.30
		0.25	24	76	1.60	0.80
		0.25	24	50	1.60	0.80
		0.25	24	2	0.40	0.30
	50	0.25	52	58	1.60	0.80
		0.25	52	39	1.60	0.80
		0.25	24	76	1.60	0.80
		0.25	24	50	1.60	0.80
325	24	0.60	52	58	1.60	0.80
		0.60	52	39	0.30	0.20
		0.60	52	2	0.30	0.20
		0.60	24	76	0.50	0.40
	24	0.60	24	50	0.50	0.40
		0.60	24	2	0.30	0.20

*Material passed through the sieve indicated.

Table 8. Results of minimum ignition energy tests under controlled environmental air conditions
(cont'd)

Sample	U.S. sieve number*	Concentration (gm/l)	Temperature °C	Relative humidity (%)	Minimum ignition energy	
					60 (joule)	No Go (joule)
Comp-B	100	0.30	52	58	4.80	4.00
		0.30	52	39	4.00	3.20
		0.30	52	2	0.50	0.40
		0.30	24	76	4.80	4.00
		0.30	24	50	4.00	3.20
		0.30	24	2	2.40	1.60
200	0.40	52	58	2.40	1.60	
	0.40	52	39	1.60	0.80	
	0.40	52	2	0.30	0.20	
	0.40	24	76	1.60	0.80	
	0.40	24	50	1.60	0.80	
	0.40	24	2	0.30	0.20	
325	0.45	52	58	2.40	1.60	
	0.45	52	39	1.60	0.80	
	0.45	52	2	0.30	0.20	
	0.45	24	76	1.60	0.80	
	0.45	24	50	0.80	0.50	
	0.45	24	2	0.20	0.10	

*Material passed through the sieve indicated.

Table 8. Results of minimum ignition energy tests under controlled environmental air conditions
(cont'd)

Sample	U.S. sieve number ^a	Concentration (gm/l)	Temperature °C	Relative humidity (%)	Minimum ignition energy	
					Gc (joule)	No Go (joule)
HMX	Class 1b	2.00	52	58	7.20	6.40
		2.00	52	39	4.80	4.00
		2.00	52	2	2.40	1.60
		2.00	24	76	6.40	5.60
		2.00	24	50	4.80	4.00
		2.00	24	25	3.20	2.40
		2.00	24	2	1.60	0.80
Class 3	60	2.00	52	58	>8.00 ^c	8.00
		2.00	52	39	>8.00	8.00
		2.00	52	2	3.20	2.40
		2.00	24	76	>8.00	8.00
		2.00	24	50	6.40	5.60
		2.00	24	2	>8.00	8.00
Class 5	60	2.00	52	58	8.00	7.20
		2.00	52	39	7.20	6.40
		2.00	52	2	>8.00	8.00
		2.00	24	76	>8.00	8.00
		2.00	24	50	>8.00	8.00
		2.00	24	2	8.00	7.20

^aMaterial passed through the sieve indicated.

^bTable 22 defines class particle size designations.

^cThe test apparatus could not discharge more than 8.00 joules.

Table 9. Results of minimum ignition energy tests at reduced dust concentrations

Sample	U.S. sieve number ^b	Concentration (gm/l)	Min. ign. energy ^a	
			Go (joule)	No Go (joule)
M-1	100	0.15	1.60	0.80
		0.05	>8.00 ^c	8.00
	200	0.12	0.30	0.20
		0.04	7.20	6.40
	325	0.18	0.20	0.10
		0.06	2.40	1.60
M-30	100	0.21	3.20	2.40
		0.07	>8.00	8.00
	200	0.15	3.20	2.40
		0.05	>8.00	8.00
	325	0.36	0.80	0.50
		0.12	1.60	0.80
Comp R	100	0.18	2.40	1.60
		0.06	>8.00	8.00
	200	0.24	0.20	0.10
		0.08	2.40	1.60
	325	0.27	0.20	0.10
		0.09	0.80	0.50
HMX	Class 1	1.50	2.40	1.60
		0.50	6.40	5.60
	Class 3	1.50	>8.00	8.00
		0.50	>8.00	8.00
	Class 5	1.50	>8.00	8.00
		0.50	>8.00	8.00

^aAll data was obtained using 240C test air containing 1% relative humidity.

^bMaterial passed through the sieve indicated.

^cThe test apparatus could not discharge more than 8.00 joules.

Table 10. Summary of ambient minimum concentrations,
energy and temperature test results

Sample	Sieve No.	Min. expl. conc. (gm/l)	Min. ign. en.		Min. ign. temp.	
			Conc. (gm/l)	Energy (joule)	Conc. (gm/l)	Temp. (°C)
M-1	100	0.05	0.25	0.25	0.46	220
	200	0.04	0.20	0.20	0.37	230
	325	0.06	0.30	0.20	0.37	240
			0.25	0.20	0.27	240
					0.55	210
					0.46	210
M-30	100	0.07	0.50	0.50	0.92	250
	200	0.05	0.35	0.40	0.64	250
	325	0.12	0.25	0.25	0.92	230
			0.60	0.25	0.64	240
					0.92	230
					0.64	230
Comp B	100	0.06	0.50	0.45	0.92	390
	200	0.08	0.30	1.60	0.55	330
	325	0.09	0.25	1.60	0.46	430
			0.40	0.20	0.92	360
			0.35	0.20	0.73	350
					0.64	380
					0.46	430
			0.45	0.20	0.92	360
			0.40	0.20	0.83	330
					0.73	360
HMX	Class 1	0.50	2.00	0.45	1.38	380
	Class 3	0.50	2.00	5.60	0.92	350
	Class 5	1.00	2.00	4.00	1.38	690
					0.92	>750*
					1.38	680
					0.92	>750

*The Godbert-Greenwald furnace could not be heated past 750°C.

Table 11. Summary of ignition energy tests,
controlled environment

Sieve No.	Temp. (°C)	R.H. (%)	Minimum ignition energy ^a			
			M-1 (joule)	M-30 (joule)	Comp B (joule)	HMX ^b (joule)
100	52	58	1.60	3.20	4.80	7.20
	52	39	0.40	2.40	4.00	4.80
	52	2	0.40	1.60	0.50	2.40
	24	76	0.40	2.40	4.80	6.40
	24	50	0.40	1.60	4.00	4.80
	24	2	0.20	0.50	2.40	1.60
200	52	58	0.40	1.60	2.40	>8.00 ^c
	52	39	0.30	1.60	1.60	>8.00
	52	2	0.20	0.40	0.30	3.20
	24	76	0.80	1.60	1.60	>8.00
	24	50	0.80	1.60	1.60	6.40
	24	2	0.30	0.40	0.30	>8.00
325	52	58	0.50	1.60	2.40	8.00
	52	39	0.40	0.30	1.60	7.20
	52	2	0.20	0.30	0.30	>8.00
	24	76	0.50	0.50	1.60	>8.00
	24	50	0.40	0.50	0.80	>8.00
	24	2	0.20	0.30	0.20	8.00

^aSee Table 8 for dust concentrations.

^bSieve numbers 100, 200 and 325 are replaced by Classes 1, 3 and 5 for HMX.

^cThe test apparatus could not discharge more than 8 joules.

Table 12. Summary of ignition energy tests, reduced dust concentrations

Sieve No.	Minimum ignition energy ^a						HMX ^b	
	M-1		M-30		Comp B			
	Conc. (gm/l)	Energy (joule)	Conc. (gm/l)	Energy (joule)	Conc. (gm/l)	Energy (joule)		
10C	0.15	1.60	0.21	3.20	0.18	2.40	1.50	
	0.05	>8.00 ^c	0.07	>8.00	0.06	>8.00	0.50	
200	0.12	0.30	0.15	3.20	0.24	0.20	1.50	
	0.04	7.20	0.05	>8.00	0.08	2.40	0.50	
325	0.18	0.20	0.36	0.80	0.27	0.20	>8.00	
	0.06	2.40	0.12	1.60	0.09	0.20	0.50	

^aSee Table 3 for dust concentrations.

^bSieve numbers 100, 200 and 325 are replaced by Classes 1, 3 and 5 for HMX.

^cThe test apparatus could not discharge more than 8 joules.

Table 13. Propellant particle size mass distribution

<u>Grinding tests of M-1 SP propellant</u>		
<u>Start</u>	<u>135.8 grams</u>	<u>% of 132.91</u>
Above No. 50 sieve	123.57	92.97
No. 70	6.32	4.76
No. 140	1.26	0.95
No. 200	0.41	0.31
Through No. 200	<u>1.15</u>	<u>0.86</u>
	132.91 grams	99.85%
<u>Start</u>	<u>278.46 grams</u>	<u>% of 276.41</u>
Above No. 50 Sieve	240.40	86.97
No. 70	20.42	7.39
No. 150	6.10	2.21
No. 200	2.00	0.72
Through No. 200	<u>7.49</u>	<u>2.71</u>
	276.41 grams	100.00%

Table 14. Fractional test matrix

1. $C^0 T^0 P^0 M^0 S^0$	19. $C^0 T^0 P^2 M^1 S^0$	37. $C^0 T^0 P^2 M^2 S^2$
2. $C^1 T^2 P^0 M^0 S^0$	20. $C^0 T^0 P^2 M^0 S^1$	38. $C^2 T^0 P^2 M^0 S^2$
3. $C^1 T^0 P^2 M^0 S^0$	21. $C^0 T^0 P^0 M^2 S^1$	39. $C^2 T^0 P^2 M^2 S^0$
4. $C^1 T^0 P^0 M^2 S^0$	22. $C^1 T^1 P^1 M^0 S^0$	40. $C^2 T^0 P^0 M^2 S^2$
5. $C^1 T^0 P^0 M^0 S^2$	23. $C^1 T^1 P^0 M^1 S^0$	41. $C^0 T^2 P^0 M^2 S^2$
6. $C^0 T^1 P^2 M^0 S^0$	24. $C^1 T^1 P^0 M^0 S^1$	42. $C^2 T^2 P^1 M^1 S^0$
7. $C^0 T^1 P^0 M^2 S^0$	25. $C^0 T^1 P^1 M^1 S^0$	43. $C^2 T^2 P^1 M^0 S^1$
8. $C^0 T^1 P^0 M^0 S^2$	26. $C^0 T^1 P^1 M^0 S^1$	44. $C^2 T^0 P^2 M^1 S^1$
9. $C^0 T^0 P^1 M^2 S^0$	27. $C^0 T^0 P^1 M^1 S^1$	45. $C^0 T^2 P^2 M^1 S^1$
10. $C^0 T^0 P^1 M^0 S^2$	28. $C^1 T^0 P^1 M^0 S^1$	46. $C^2 T^2 P^0 M^1 S^1$
11. $C^0 T^0 P^0 M^1 S^2$	29. $C^1 T^0 P^1 M^1 S^0$	47. $C^2 T^1 P^2 M^1 S^0$
12. $C^2 T^1 P^0 M^0 S^0$	30. $C^1 T^0 P^0 M^1 S^1$	48. $C^2 T^1 P^2 M^0 S^1$
13. $C^2 T^0 P^1 M^0 S^0$	31. $C^0 T^1 P^0 M^1 S^1$	49. $C^2 T^0 P^1 M^2 S^1$
14. $C^2 T^0 P^0 M^1 S^0$	32. $C^2 T^2 P^2 M^0 S^0$	50. $C^0 T^2 P^1 M^2 S^1$
15. $C^2 T^0 P^0 M^0 S^1$	33. $C^2 T^2 P^0 M^2 S^0$	51. $C^2 T^1 P^0 M^2 S^1$
16. $C^0 T^2 P^1 M^0 S^0$	34. $C^2 T^2 P^0 M^0 S^2$	52. $C^2 T^1 P^1 M^2 S^0$
17. $C^0 T^2 P^0 M^1 S^0$	35. $C^0 T^2 P^2 M^2 S^0$	53. $C^2 T^1 P^1 M^0 S^2$
18. $C^0 T^2 P^0 M^0 S^1$	36. $C^0 T^2 P^2 M^0 S^2$	54. $C^2 T^0 P^1 M^1 S^2$

Table 14. Fractional test matrix
(cont'd)

55. $C^0 T^2 P^1 M^1 S^2$	64. $C^1 T^0 P^2 M^2 S^1$	73. $C^1 T^2 P^1 M^1 S^1$
56. $C^2 T^1 P^0 M^1 S^2$	65. $C^0 T^1 P^2 M^2 S^1$	74. $C^1 T^1 P^2 M^1 S^1$
57. $C^1 T^2 P^1 M^2 S^0$	66. $C^1 T^2 P^0 M^2 S^1$	75. $C^1 T^1 P^1 M^2 S^1$
58. $C^1 T^2 P^1 M^0 S^2$	67. $C^1 T^1 P^2 M^2 S^0$	76. $C^1 T^1 P^1 M^1 S^2$
59. $C^1 T^0 P^2 M^1 S^2$	68. $C^1 T^1 P^2 M^0 S^2$	77. $C^2 T^2 P^2 M^2 S^1$
60. $C^0 T^1 P^2 M^1 S^2$	69. $C^1 T^0 P^1 M^2 S^2$	78. $C^2 T^1 P^2 M^2 S^2$
61. $C^1 T^2 P^0 M^1 S^2$	70. $C^0 T^1 P^1 M^2 S^2$	79. $C^1 T^2 P^2 M^2 S^2$
62. $C^1 T^2 P^2 M^1 S^0$	71. $C^1 T^1 P^0 M^2 S^2$	80. $C^2 T^2 P^1 M^2 S^2$
63. $C^1 T^2 P^2 M^0 S^1$	72. $C^2 T^1 P^1 M^1 S^1$	81. $C^2 T^2 P^2 M^1 S^2$

Table 15. Test Case I data^a

Propellant Size: ≤ 75 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: None added

Test No.	Minimum ignition energy		
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	Result ^c
2	488	0.5	+
3	488	0.375	+
4	488	0.30	+
5	488	0.25	+
6	488	0.20	+
7	488	0.15	-
8	488	0.15	+
9	488	0.15	-
Minimum explosive concentration			
2	488	0.5	+
41	244	0.5	+
33	203	8.0	-
28	163	8.0	-
23	122	8.0	-

Conclusion: Minimum ignition energy < 0.15 joule and minimum explosive concentration lies between 203 and 244 mg/l.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 16. Test Case II data^a

Propellant Size: 75-105 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: None added

Test No.	<u>Minimum ignition energy</u>		
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	Result ^c
49	488	0.50	+
50	488	0.45	+
51	488	0.40	+
52	486	0.35	+
53	488	0.30	+
54	488	0.25	+
56	488	0.225	+
55	488	0.20	-
66	366	0.175	+
65	366	0.15	-
<u>Minimum explosive concentration</u>			
49	488	0.50	+
63	366	0.25	+
57	244	0.30	+
62	122	8.00	-

Conclusion: Minimum ignition energy <0.175 joule and minimum explosive concentration lies between 122 and 244 mg/l.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 17. Test Case III data^a

Propellant Size: 75-105 microns
Moisture Content: Up to 3% by weight (propellant not placed in desiccator)
Solvent: None added

<u>Minimum explosive concentration</u>			
<u>Test No.</u>	<u>Propellant^b concentration (mg/l)</u>	<u>Ignition energy (Joule)</u>	<u>Result^c</u>
71	366	8.0	-
70	244	8.0	-
69	163	8.0	-
68	122	8.0	-

Conclusion: Minimum dust concentration is above 366 mg/l.

^aAir reservoir pressure in all test cases was 60 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 18. Test Case IV data^a

Propellant Size: 75-105 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: Propellant consisted of 10% ethyl alcohol by weight

Test No.	<u>Minimum ignition energy</u>		
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	Result ^c
79	366	8.0	-
78	366	2.0	-
<u>Minimum explosive concentration</u>			
79	366	8.0	-
77	244	8.0	-
74	122	8.0	-

Conclusion: Insufficient data for conclusions.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 19. Test Case V data^a

Propellant Size: <75 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: Propellant consisted of 10% ethyl alcohol by weight

Test No.	Minimum ignition energy		Result ^c
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	
92	366	2.0	-
<u>Minimum explosive concentration</u>			
92	366	2.0	-
91	244	8.0	-
82	122	8.0	-

Conclusion: Insufficient data for conclusions.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 20. Test Case VI data^a

Propellant Size: 75-105 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: Propellant consisted of 10% anhydrous ether by weight

Test No.	<u>Minimum ignition energy</u>		
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	Result ^c
100	366	8.0	-
99	366	2.0	-
<u>Minimum explosive concentration</u>			
100	366	8.0	-
98	244	8.0	-
95	122	8.0	-

Conclusion: Insufficient data for conclusions.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 21. Test Case VII data^a

Propellant Size: <75 microns
 Moisture Content: 0% by weight (propellant placed in desiccator)
 Solvent: Propellant consisted of 10% anhydrous ether by weight

Test No.	<u>Minimum ignition energy</u>		Result ^c
	Propellant ^b concentration (mg/l)	Ignition energy (joule)	
107	366	2.0	-
108	366	8.0	-
<u>Minimum explosive concentration</u>			
108	366	8.0	-
106	244	8.0	-
103	122	8.0	-

Conclusion: Insufficient data for conclusions.

^aAir reservoir pressure in all test cases was 68 kPa. Spark gap width was 0.318 cm. Delay time between air reservoir release and spark discharge was about 0.5 sec in all test cases.

^bThe mass of the propellant placed into the Hartmann Apparatus was divided by 1.23 liters, the volume of the Hartmann Apparatus, to obtain concentration of the dispersed dust.

^cFor minimum ignition energy tests, (+) denotes ignition of dust, while (-) denotes failure to ignite. Similarly, for minimum explosive concentration tests, (+) indicates that an explosion occurred, while (-) indicates failure to do so. Criteria for determination of (+) or (-) were the same as those listed in Bureau of Mines Report 5624. These criteria have been previously described in this report.

Table 22. Composition of M-1 propellant and ball powder

	<u>M-1 propellant</u>	<u>Ball powder</u>
Nitrocellulose (13% N)	84.2	97.7
2 - Nitrodiphenylamine	-	1.3
Dinitrotoluene	9.9	-
Dibutylphthalate	4.9	-
Diethylphthalate	-	1.0
Dephenylamine	1.0	-
Moisture and volatiles*	1.25	1.0

*Not normally included in composition specification values, but considered an added ingredient.

Table 23. Ignition characteristics of HMX dusts^a

<u>Size</u>	Concentration ^b (g/liter)			
	<u>1.5</u>	<u>0.75</u>	<u>0.3</u>	<u>0.15</u>
177 - 250 μ	N	N	N	N
125 - 177 μ	F	F	I	N
88 - 125 μ	F	F	F	N

^aIgnition energy = 3 joules

^bN = No flame propagation

F = Ignition followed by flame propagation

I = Inconsistent ignition

Table 24. Ignition characteristics of RDX dusts^a

<u>Size</u>	<u>Concentration^b</u> (g/liter)			
	<u>1.5</u>	<u>0.75</u>	<u>0.3</u>	<u>0.15</u>
177 - 250 μ	N	N	N	N
125 - 177 μ	F	N	N	N
88 - 125 μ	F	F	N	N
63 - 88 μ	F	F	F	N

^aIgnition energy = 3 joules

^bSome clumping of material

N = No flame propagation

F = Ignition followed by flame propagation

Table 25. Ignition characteristics of ball powder and M-1

<u>Compound</u>	<u>Concentration</u> (g/liter)	<u>Particle</u> <u>size</u> (μ m)	<u>Ignition</u> <u>Characteristics</u> ^a
M-1 propellant	0.1	75-150	No ignition
	0.06	44- 75	Ignition but flame fails to propagate
	0.3	44-150	Ignition and flame propagation
Ball powder	3.0	<u>Size</u> <u>Distribution</u> ^b	Slow burning
		<u>F</u> <u>A (mm)</u>	
		0.11 20-30	
		0.45 30-50	
		0.28 50-70	
		0.04 70-100	

^aIgnition energy = 3 joules

^bF = Fraction of particles within a given size range

Δ = Size range of particles within a group

Table 26. Sampling at Radford Army Ammunition Plant

<u>Operation</u>	<u>Sampling Time (in minutes)</u>	<u>Concentration (g/liter)</u>
Tray dumping at AHH (at top of bin - vented on two of four sides)	2	$<18.3 \times 10^{-6}$
Blending of ingredients for N-5 roll powder (at mouth of blending barrel - one top and one bottom)	5	$<7.4 \times 10^{-6}$
	5	$<12.4 \times 10^{-6}$
Grinding of 2 UNPA and KNO_3 (behind earthen grinder)	5	$<6.7 \times 10^{-6}$
	15	$<4.2 \times 10^{-6}$
Block breaking of nitrocellulose (at output chute of block-breaker)	8	$<4.6 \times 10^{-6}$
	8	$<7.8 \times 10^{-6}$
Packing can with M-30/105-mm propellant (at bottom level where propellant dumps into bin)	17	64.0×10^{-6}

Table 27. Sampling at Indiana Army Ammunition Plant

<u>Operation* bag loading</u>	<u>Sampling time (in hours)</u>	<u>Concentration (g/liter)</u>
5P	2	58×10^{-6}
6P	2	4×10^{-6}
7P	2-1/4	1×10^{-6}
8P	1-1/4	10×10^{-6}
9P	1-1/4	19×10^{-6}
10P	2-1/4	85×10^{-6}
12P	3-1/4	294×10^{-6}
14P	1-1/6	242×10^{-6}
15P	2	20×10^{-6}
16P	1-1/6	165×10^{-6}
17P	1-1/6	78×10^{-6}
19P	2	6×10^{-6}
20P	1-1/6	199×10^{-6}

*Details of sampling sites are given in Figure 5.

Table 28. Uncontrolled sampling at Indiana Army Ammunition Plant

<u>Sample</u>	<u>Concentration collected g/90 min/10.74 cm²</u>
2P	0.165
3P	0.016
4P	0.020
11P	0.748
21P	0.004

These samples cannot be related to a known flow rate of air, but instead relate to "fallout" over a specified area.

Table 29. Sampling at Milan Army Ammunition Plant

<u>Operation</u>	<u>Sampling time (in hours)</u>	<u>Concentration (g/liter)</u>
Comp B flake dump		
at mouth of Hood #3	3-1/4	Negligible
at mouth of Hood #2	3-1/4	Negligible
Conveyor behind Hood #3	1/2	Negligible
Conveyor behind Hood #3	4-1/3	1.92×10^{-6}
Conveyor behind Hood #2	4-5/12	1.28×10^{-6}
Riser scrap addition (above riser sump chute)	5-3/4	0.87×10^{-6}
Riser knockout		
at mouth of Hood #1	4-1/4	Negligible
at mouth of Hood #2	4-1/4	0.49×10^{-6}

Table 30. Minimum explosive dust concentration - Comparison of results

Results of this study		Results of other studies (see Reference Column below)		
Particle size (μm)	Dust conc. (g/liter)	Particle size (μm)	Dust conc. (g/liter)	Comments
HMX	88-125	0.15-0.3	65-320	0.5 Hartmann Apparatus Phase I
RDX	63-88	0.15-0.3	124-154	0.9-0.12 60/40 RDX/TNT No test for flame propagation. 35
⁸³ H-1	44-150	0.15-0.3	75-105	0.122-0.244 Hartmann Apparatus Phase II
Ball powder	See Table 2	3 g/liter failed to support flame propagation.	-	-

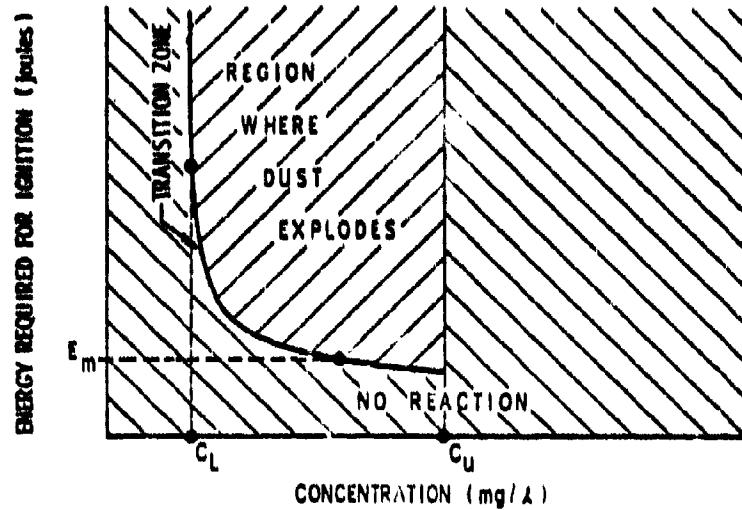


Figure 1. Typical Explosibility For A "Conventional" Flammable Dust

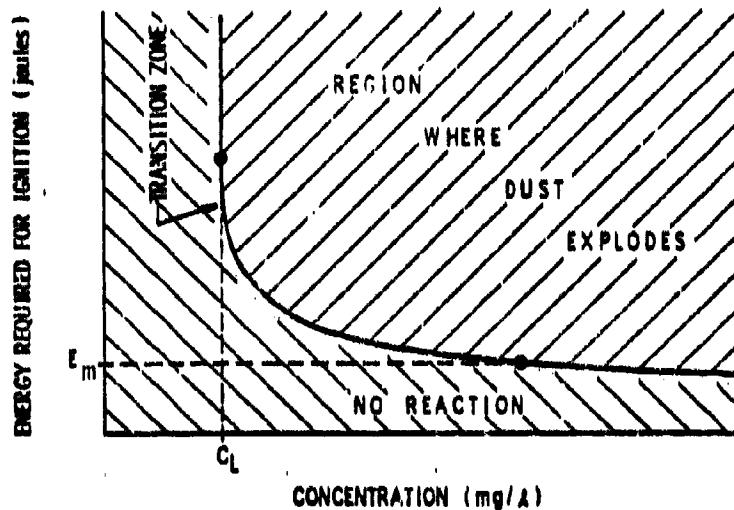


Figure 2. Anticipated Explosibility Curve For A Flammable Dust Not Requiring Oxygen

- E_m - measured minimum ignition energy
- C_L - measured minimum explosive concentration
- C_U - measured upper explosive limit for "conventional" dusts due to insufficient oxygen supply

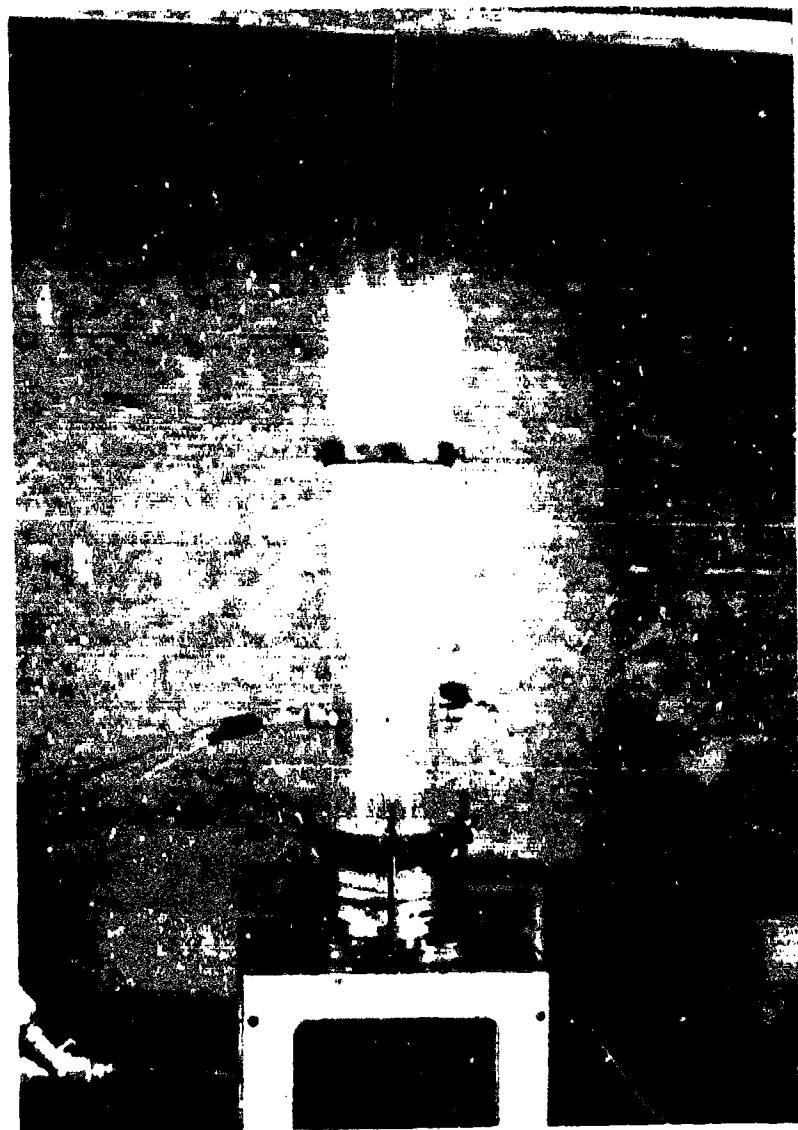


Figure 3. Dust explosion in lucite tube.

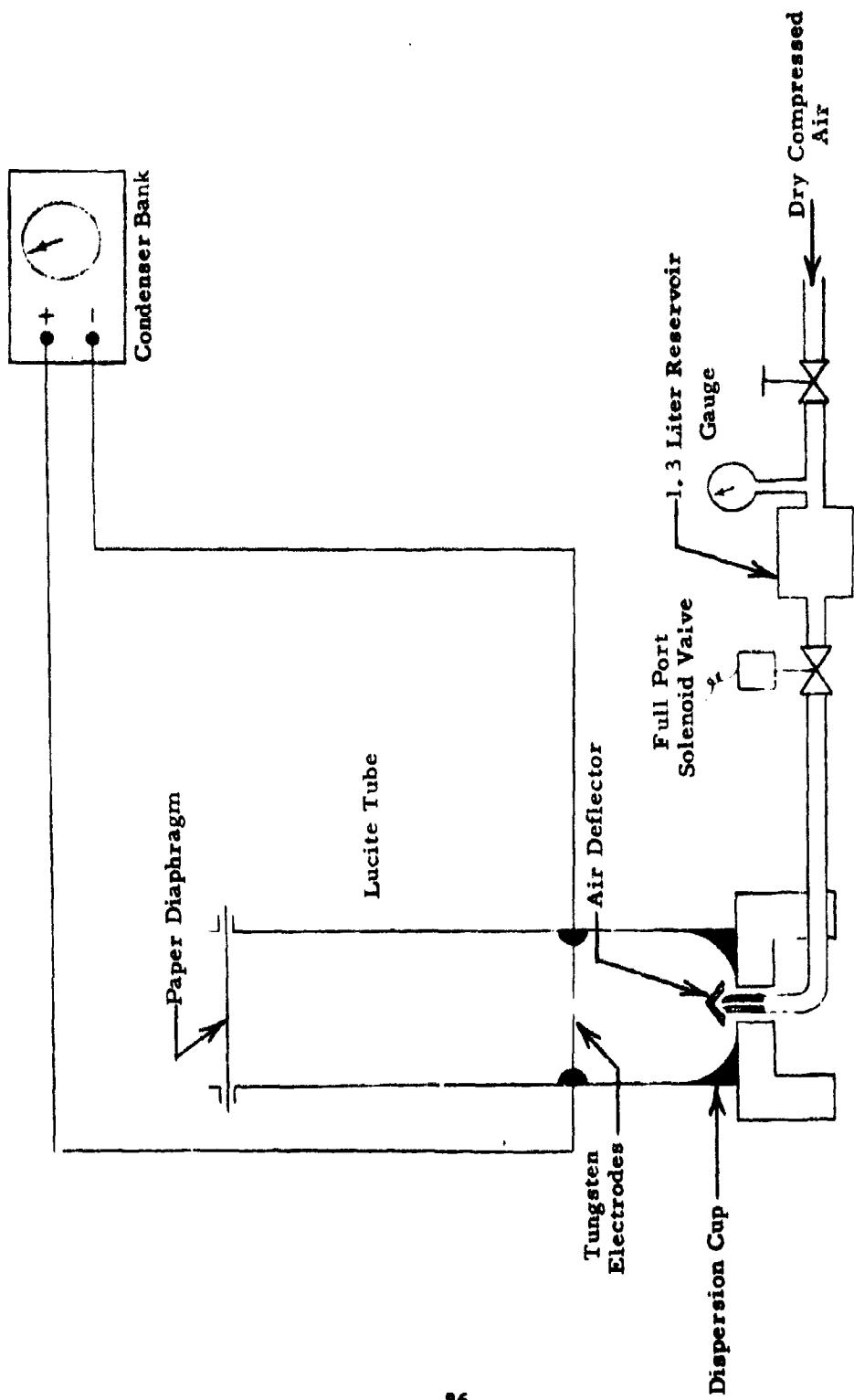


Figure 4. Hartmann apparatus -- schematic diagram.

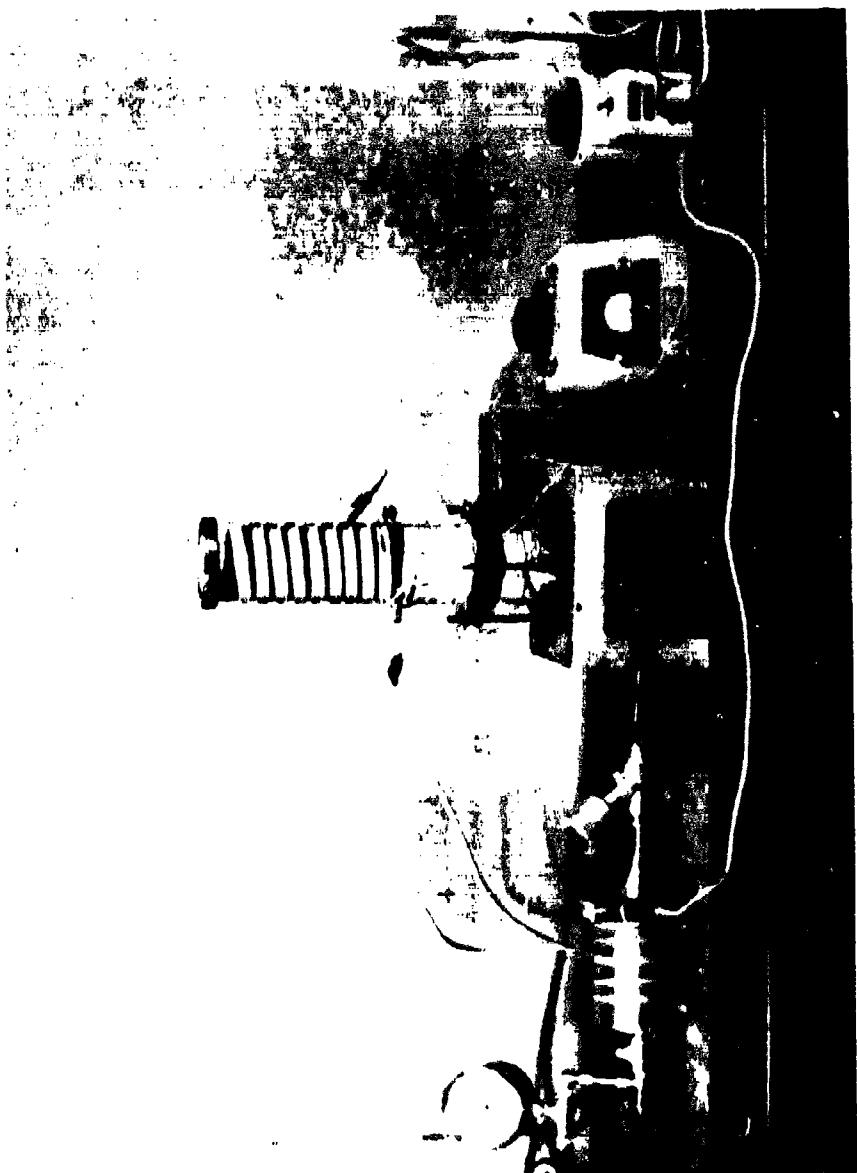


Figure 4A. Hartmann apparatus -- test setup.

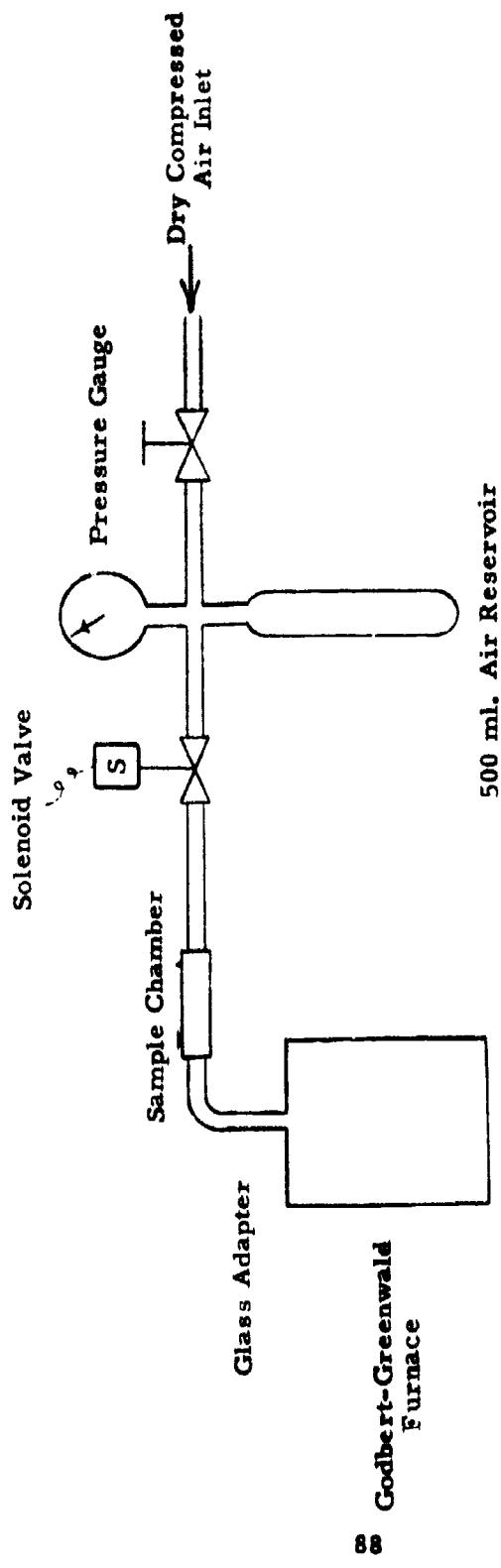


Figure 5. Schematic of minimum ignition temperature of dust cloud apparatus.

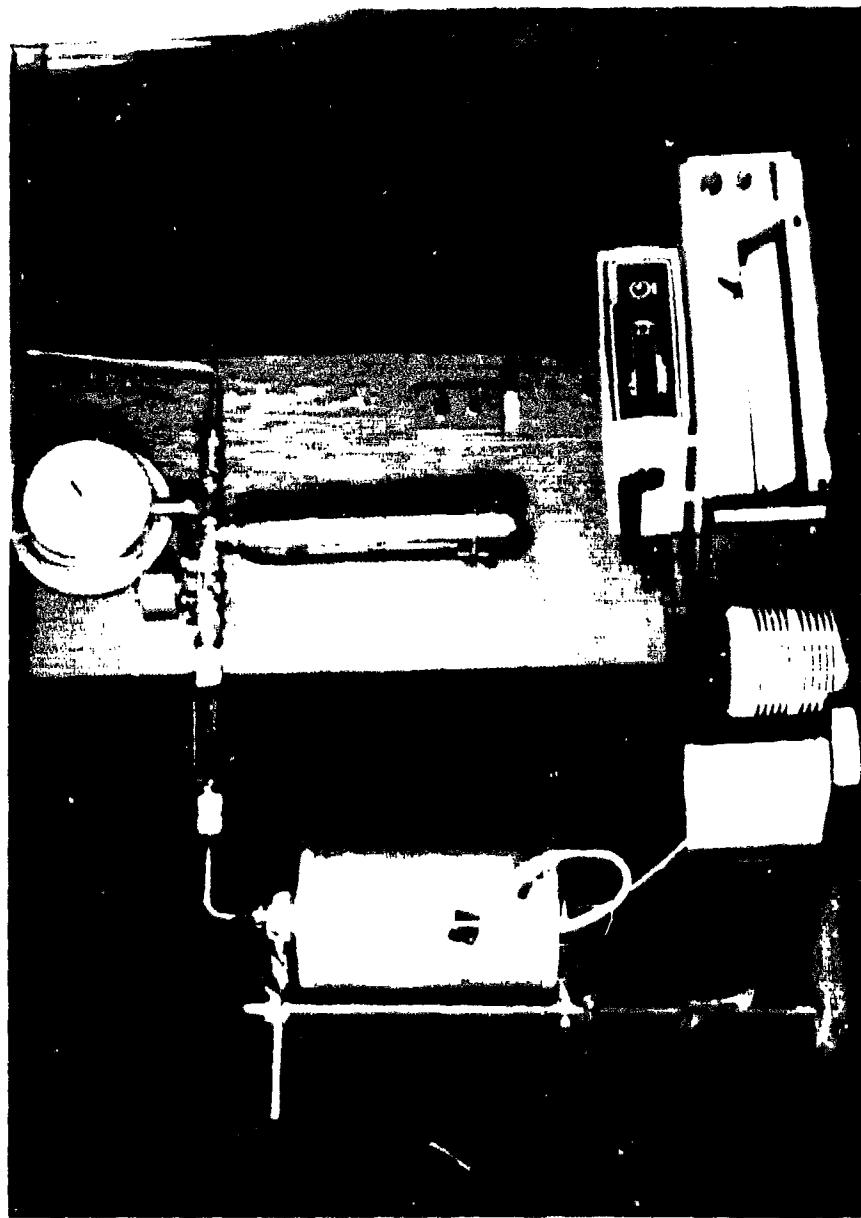


Figure 6. Minimum ignition temperature of a dust cloud apparatus.

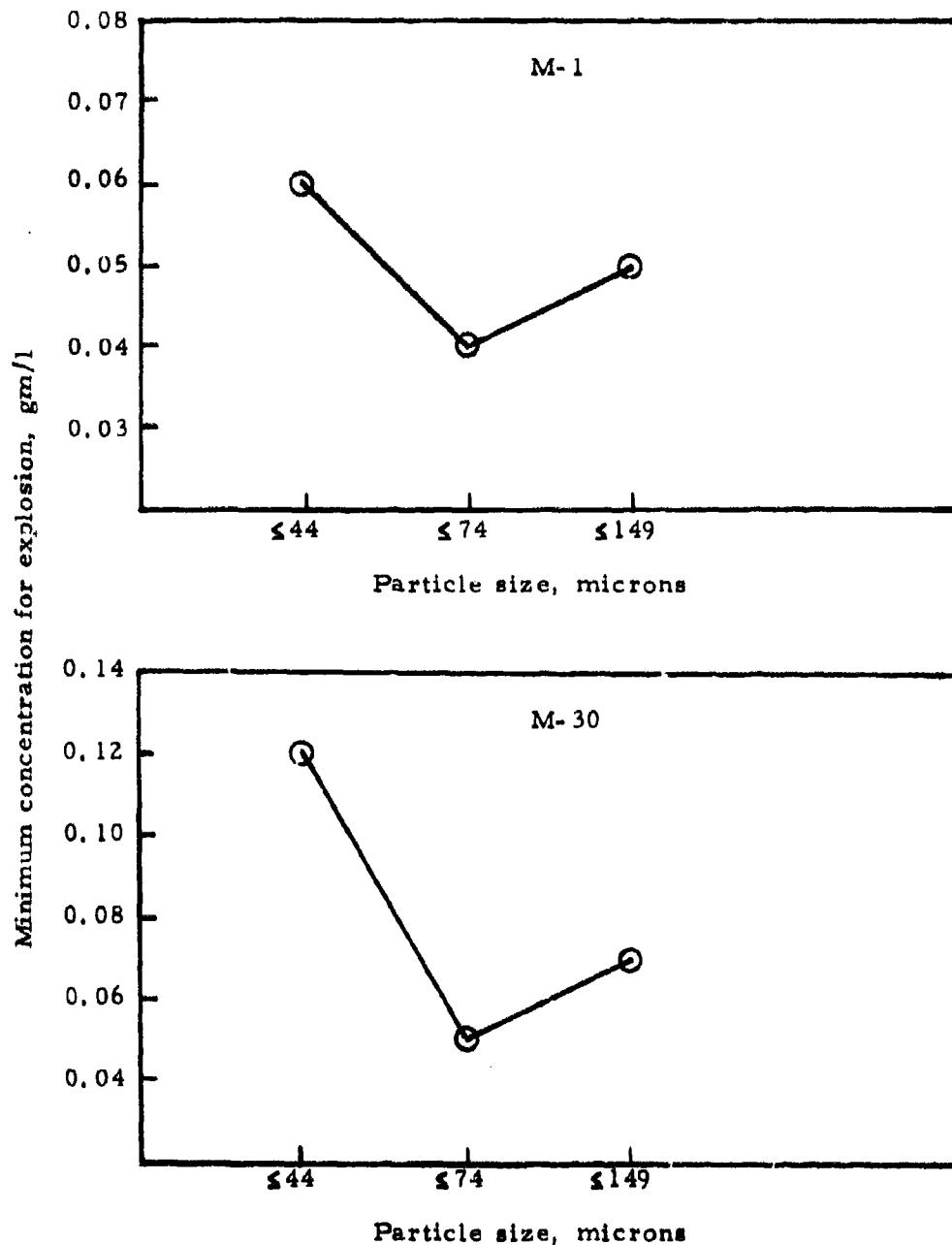


Figure 7. Minimum concentration for explosion versus particle size, M-1 and M-30 propellants.

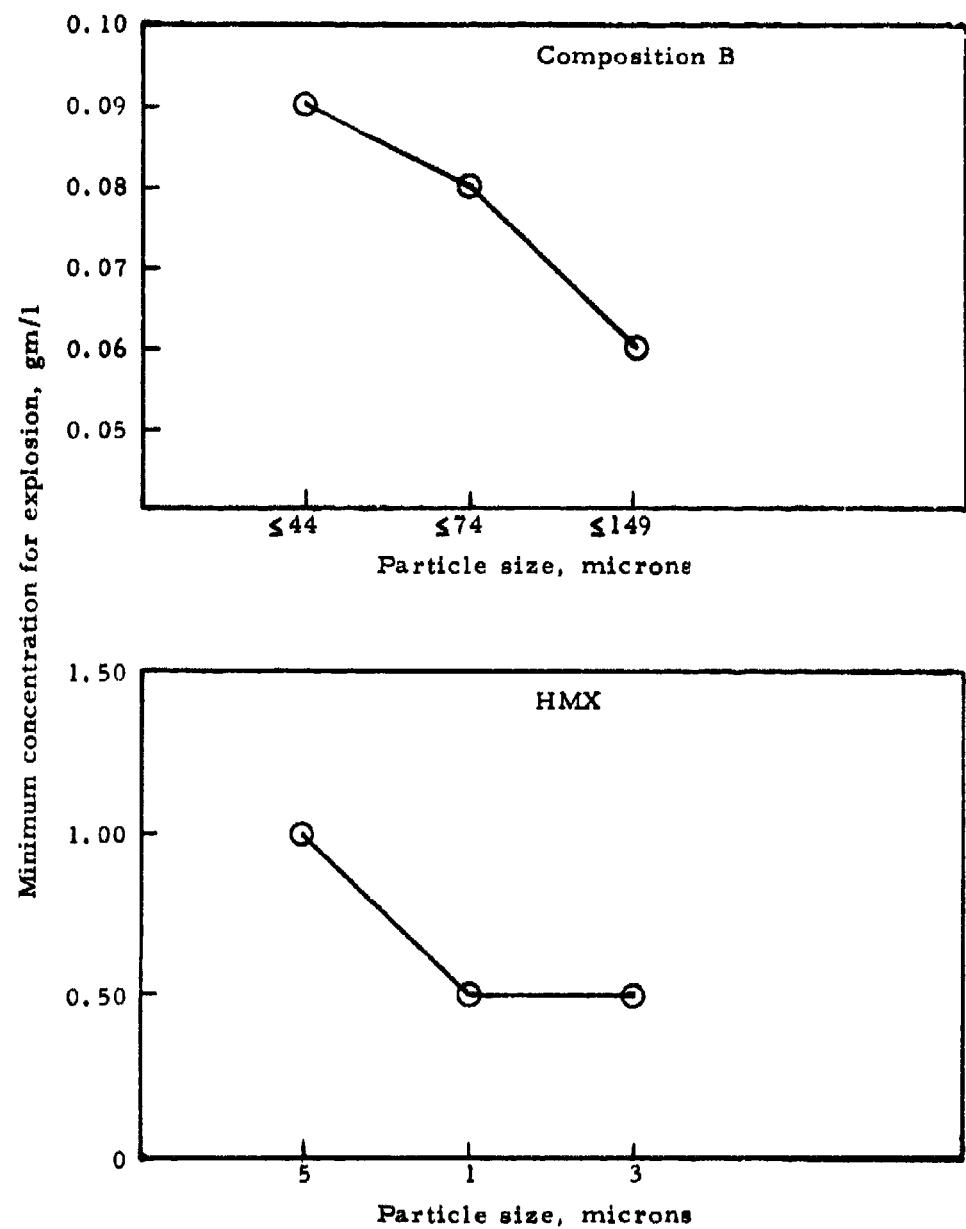


Figure 8. Minimum concentration for explosion versus particle size, Composition B and HMX explosives.

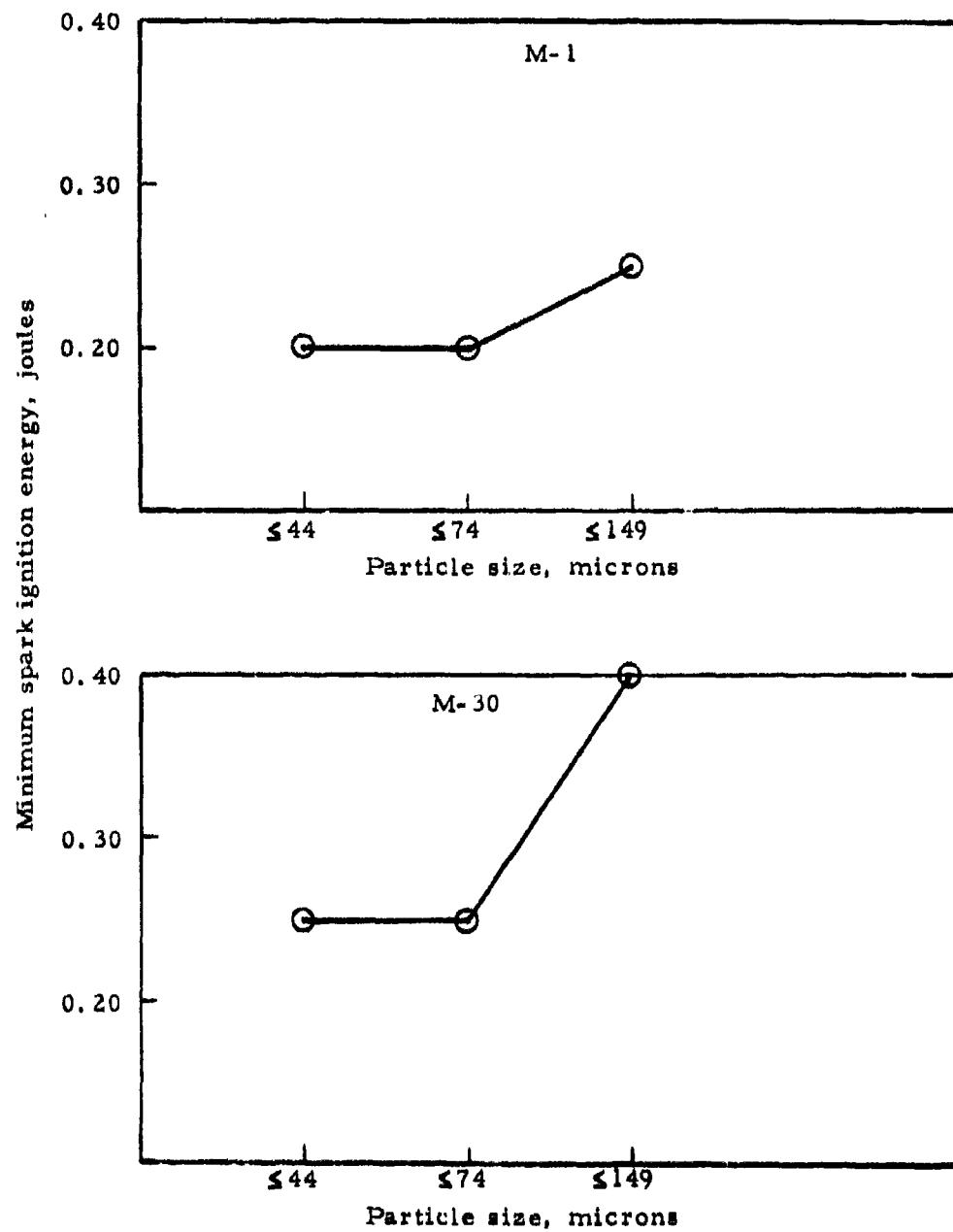


Figure 9. Minimum spark ignition energy versus particle size, M-1 and M-30 propellants.

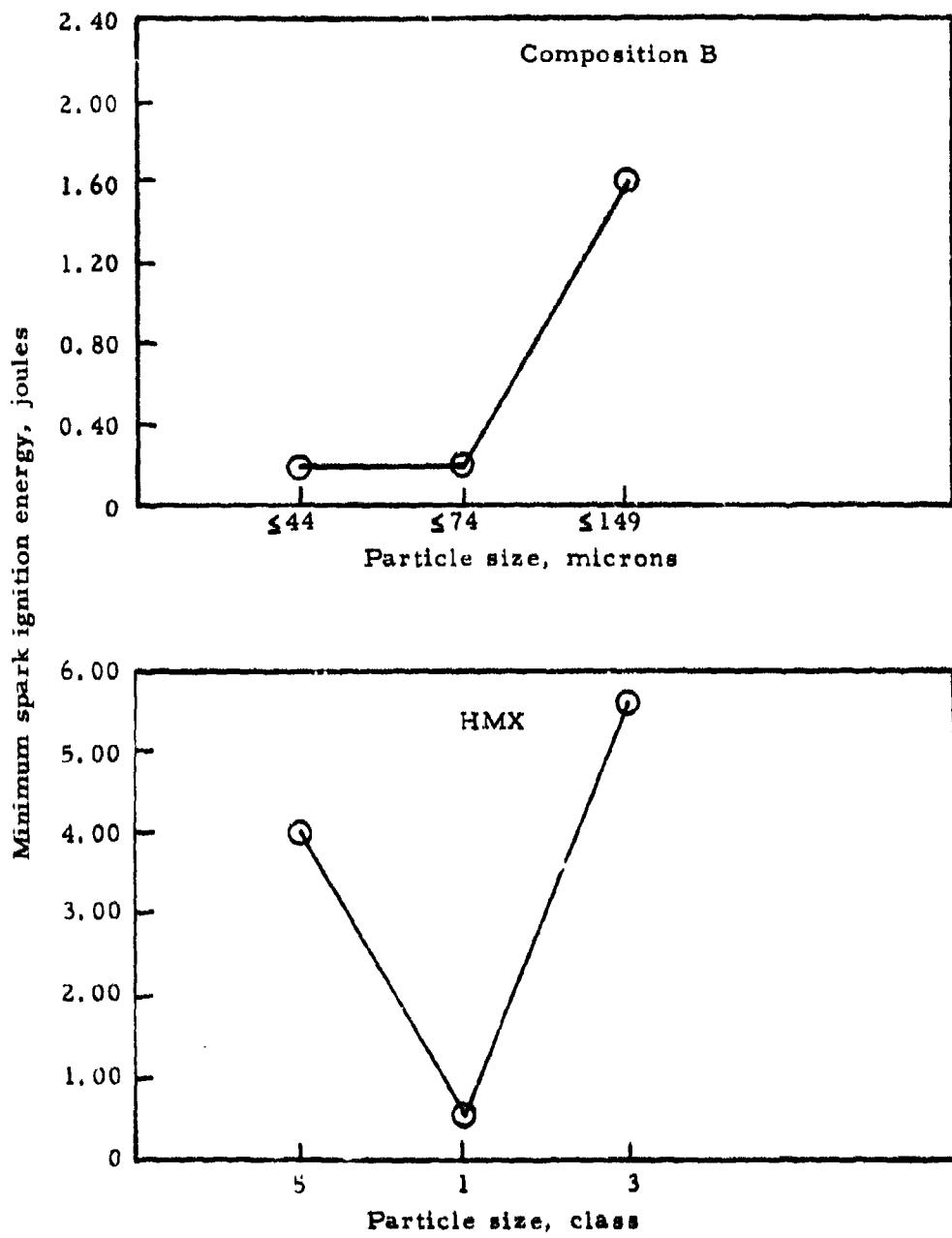


Figure 10. Minimum spark ignition energy versus particle size, Composition B and HMX explosives.

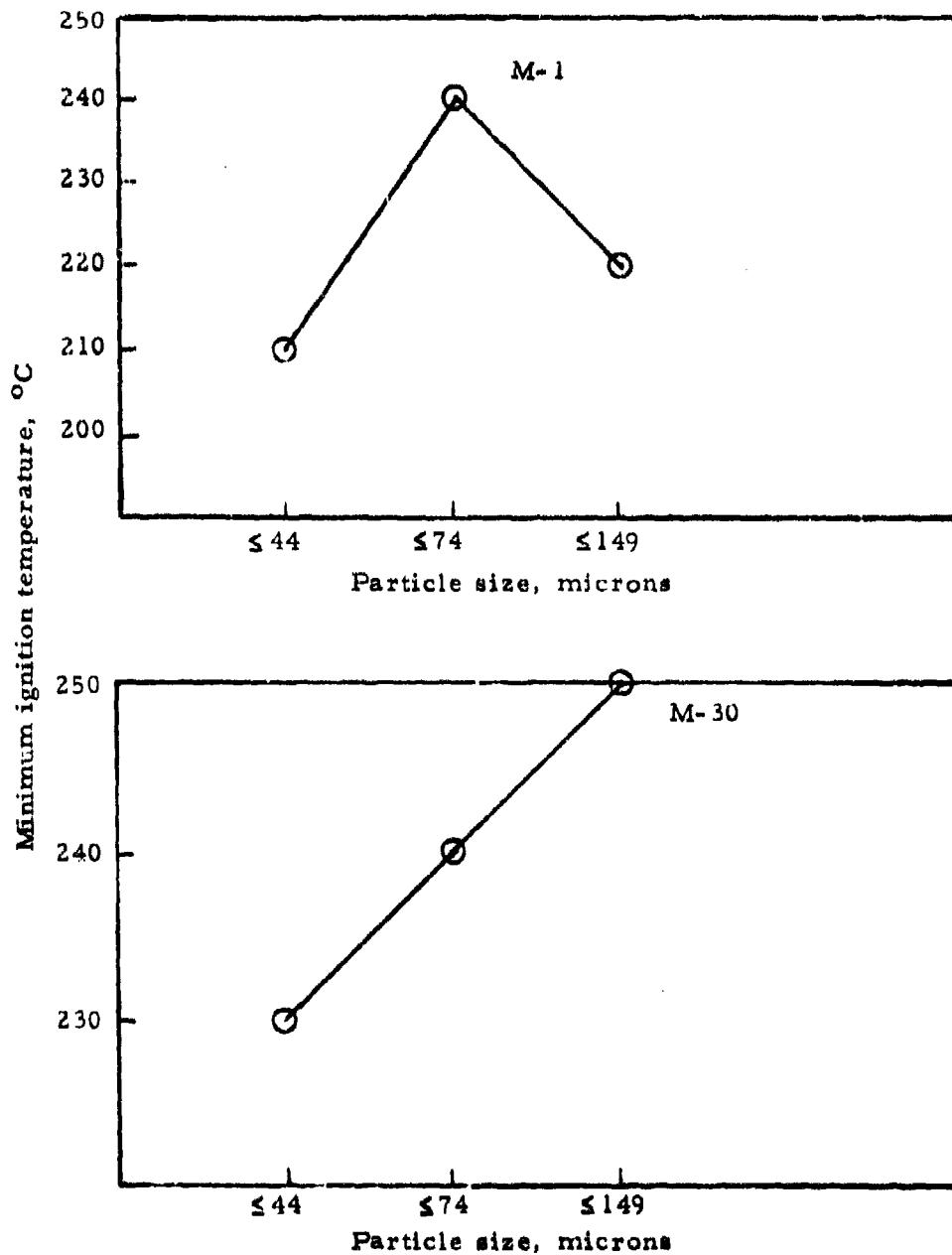


Figure 11. Minimum ignition temperature of dust clouds versus particle size, M-1 and M-30 propellants.

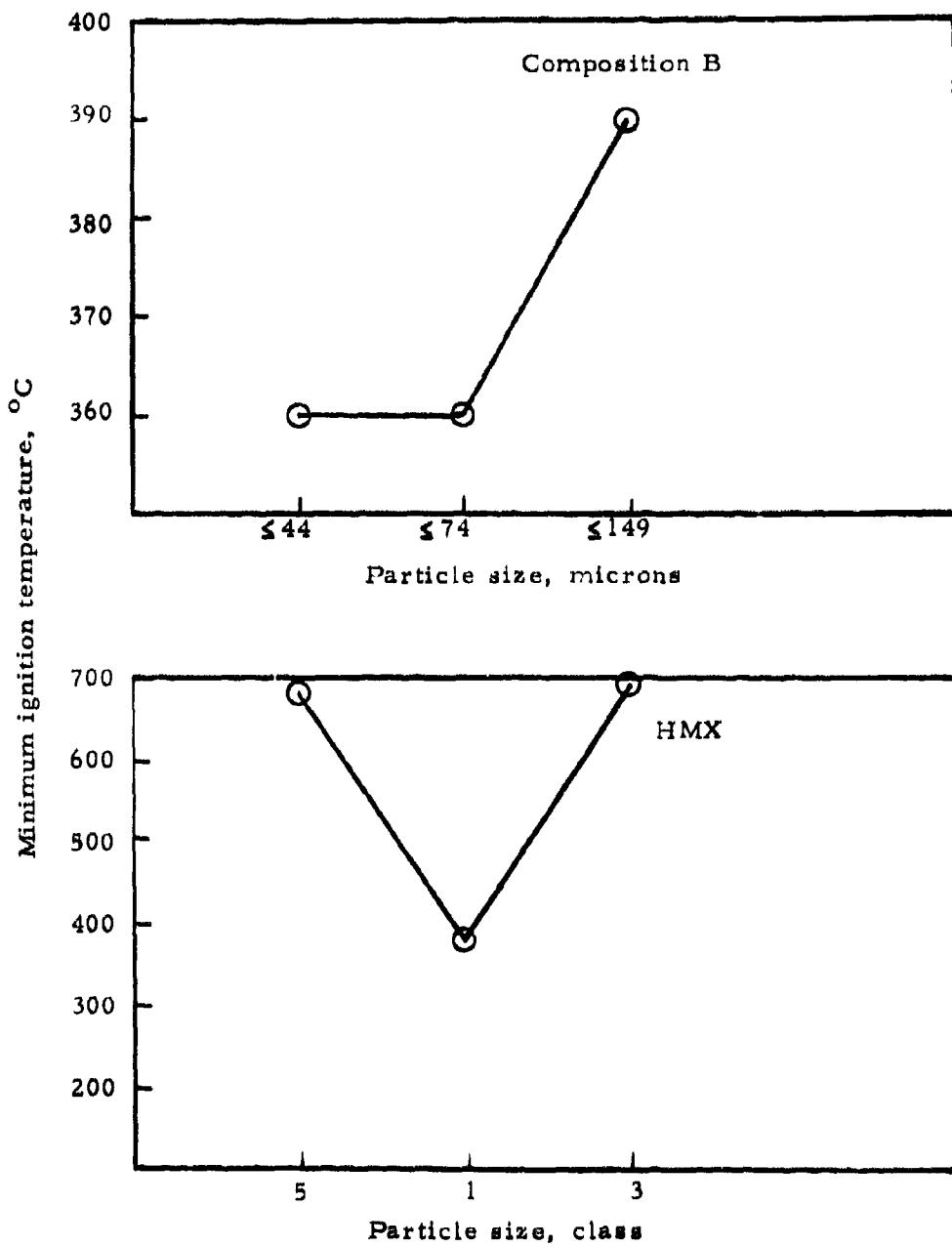


Figure 12. Minimum ignition temperature of dust clouds versus particle size, Composition B and HMX explosives.

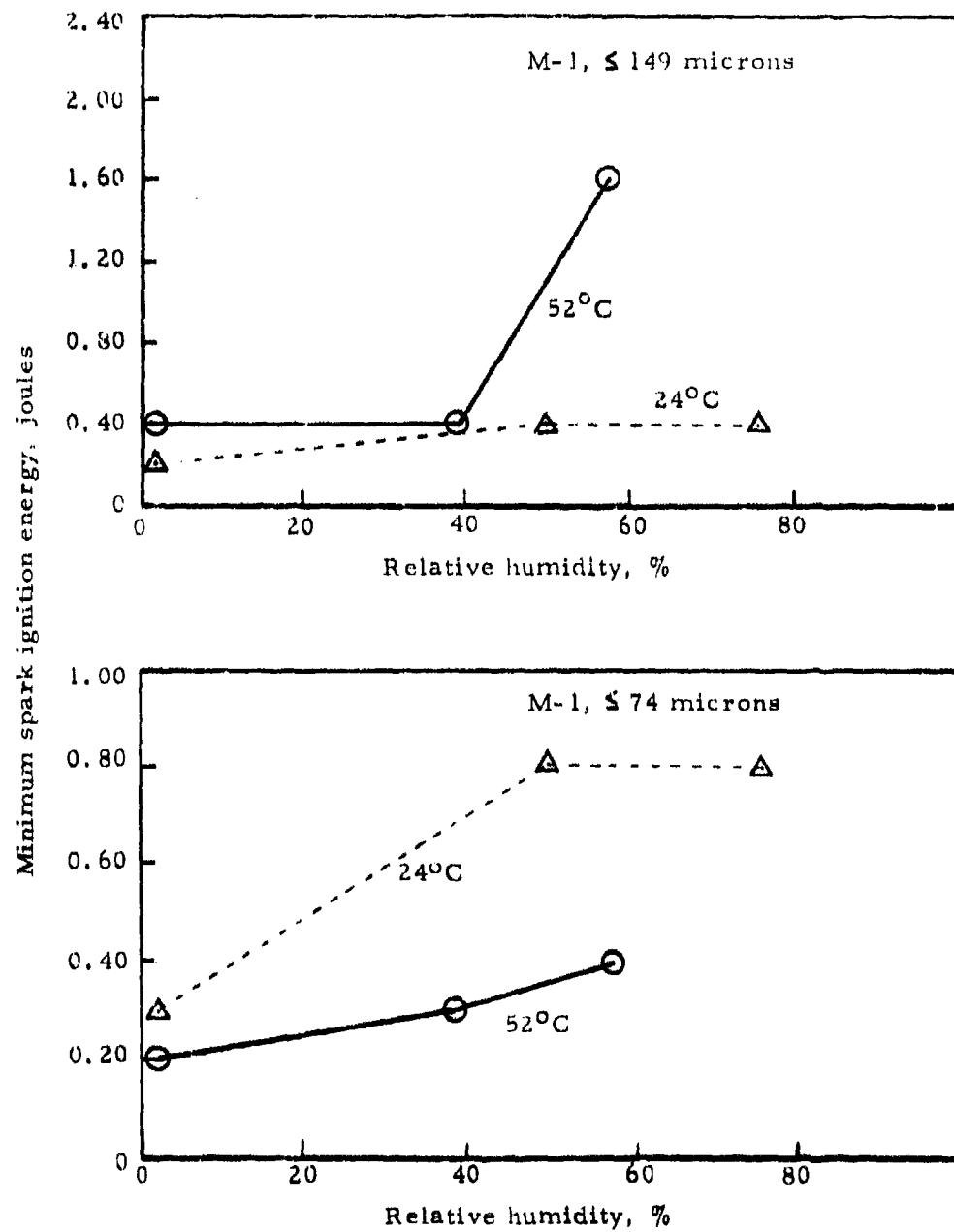


Figure 13. Minimum spark ignition energy versus relative humidity, M-1 propellant, particle size ≤ 149 and ≤ 74 microns.

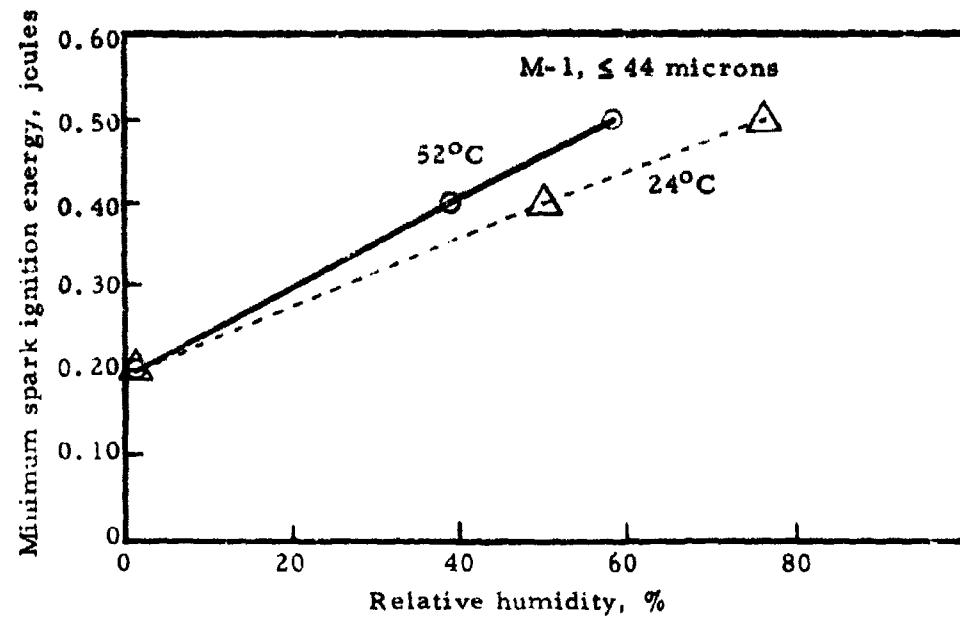


Figure 14. Minimum spark ignition energy versus relative humidity, M-1 propellant, particle size \leq 44 microns.

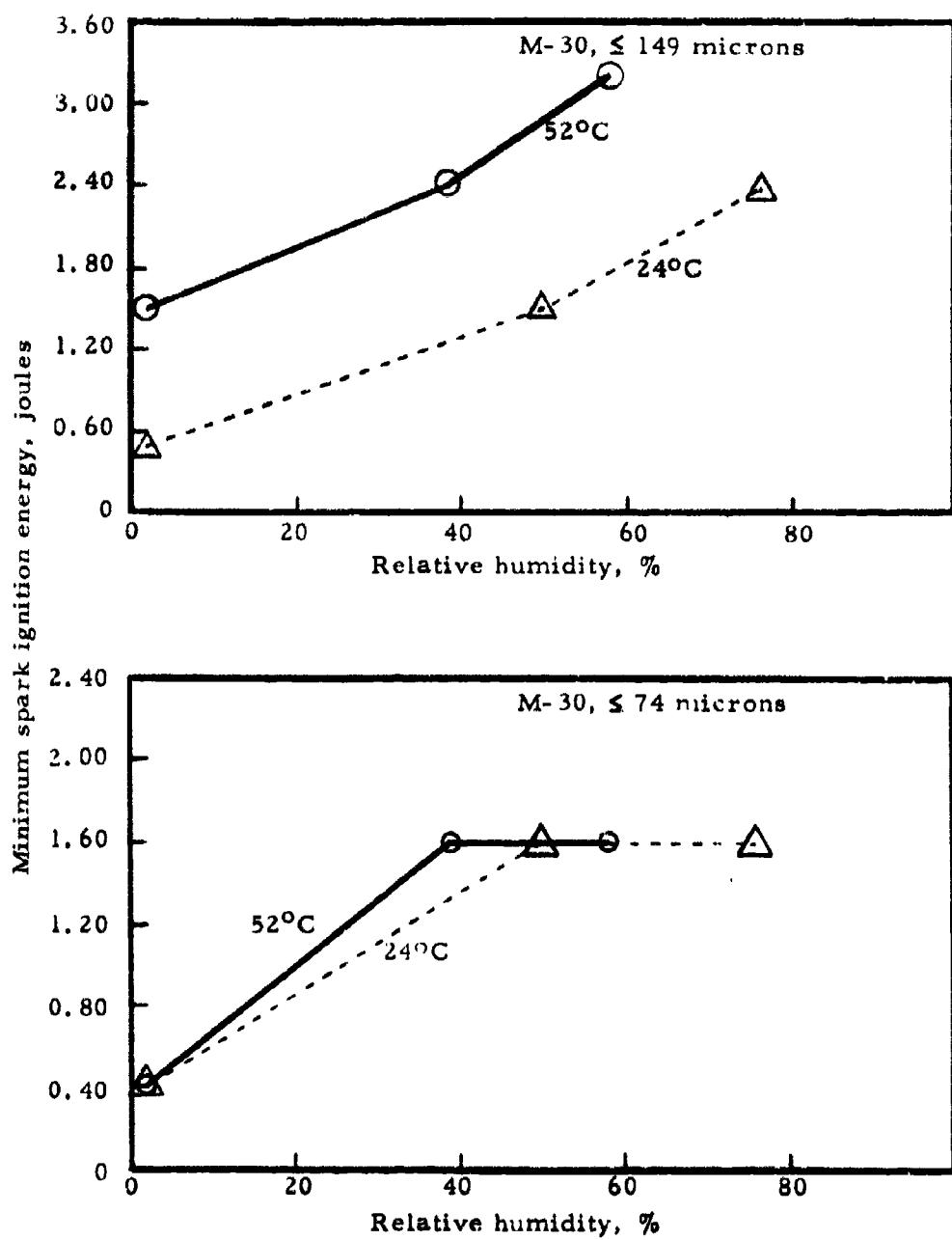


Figure 15. Minimum spark ignition energy versus relative humidity, M-30 propellant, particle size ≤ 149 and ≤ 74 microns.

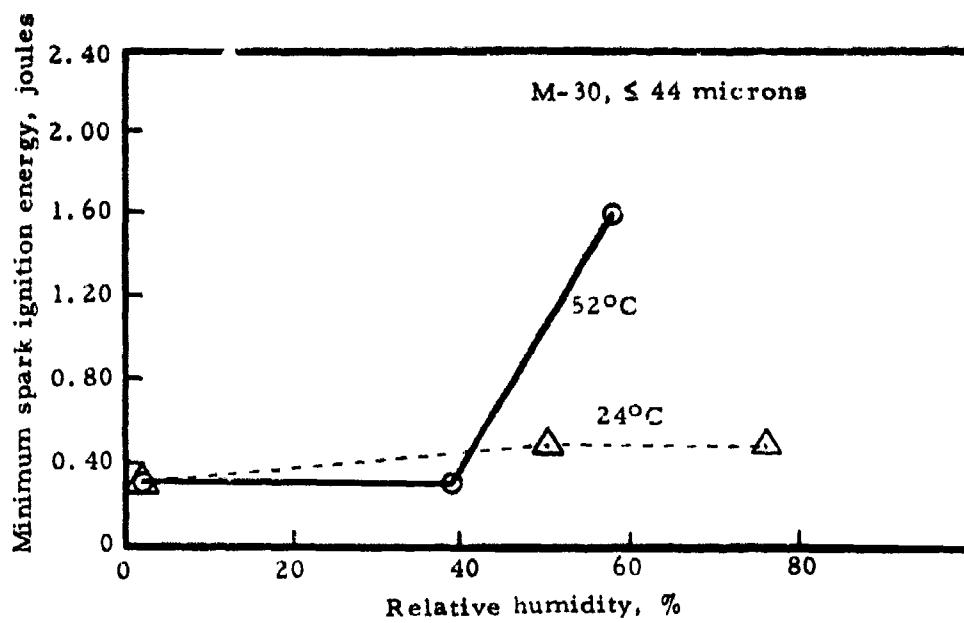


Figure 16. Minimum spark ignition energy versus relative humidity, M-30 propellant, particle size ≤ 44 microns.

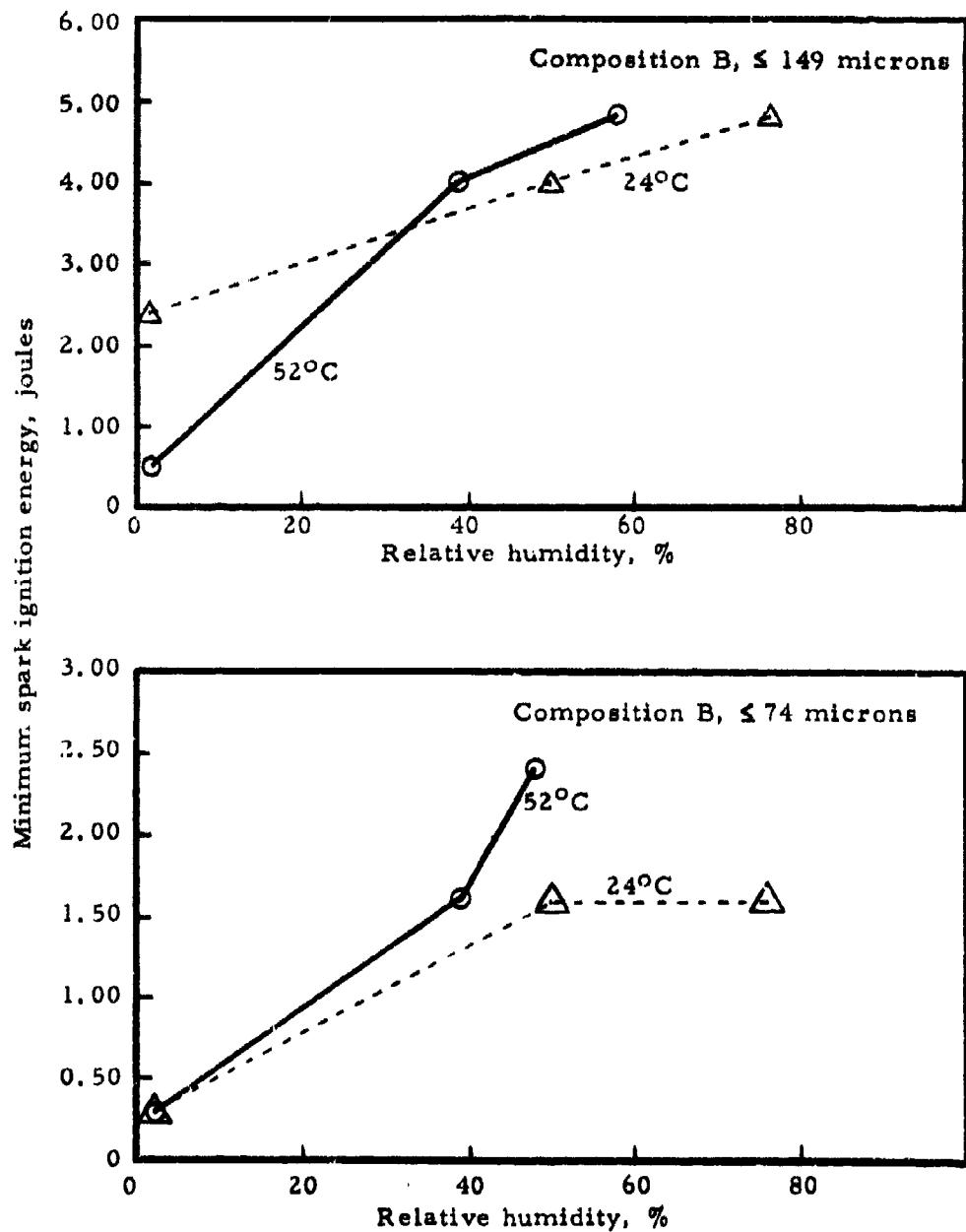


Figure 17. Minimum spark ignition energy versus relative humidity, Composition B explosive, particle size ≤ 149 and ≤ 74 microns.

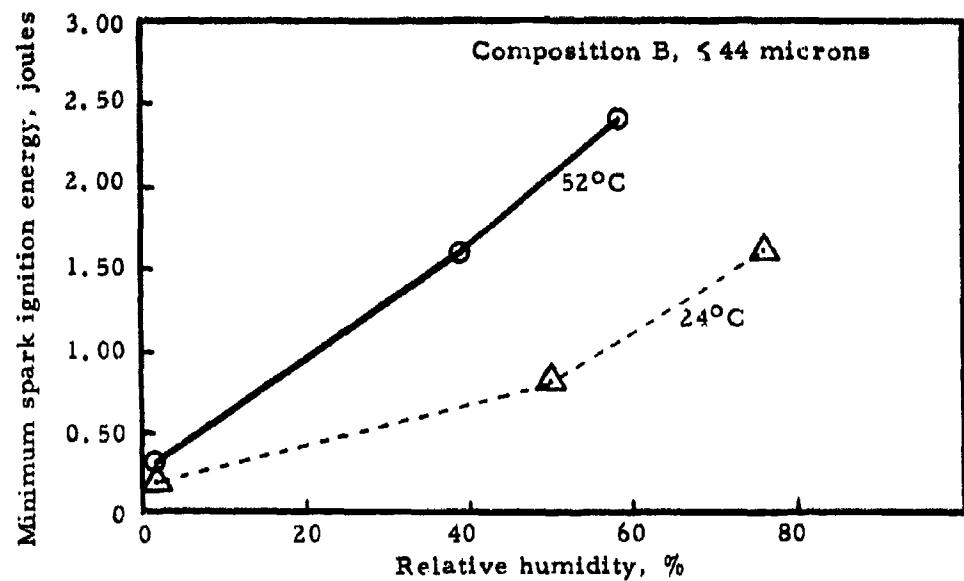


Figure 18. Minimum spark ignition energy versus relative humidity, Composition B explosive, particle size ≤ 44 microns.

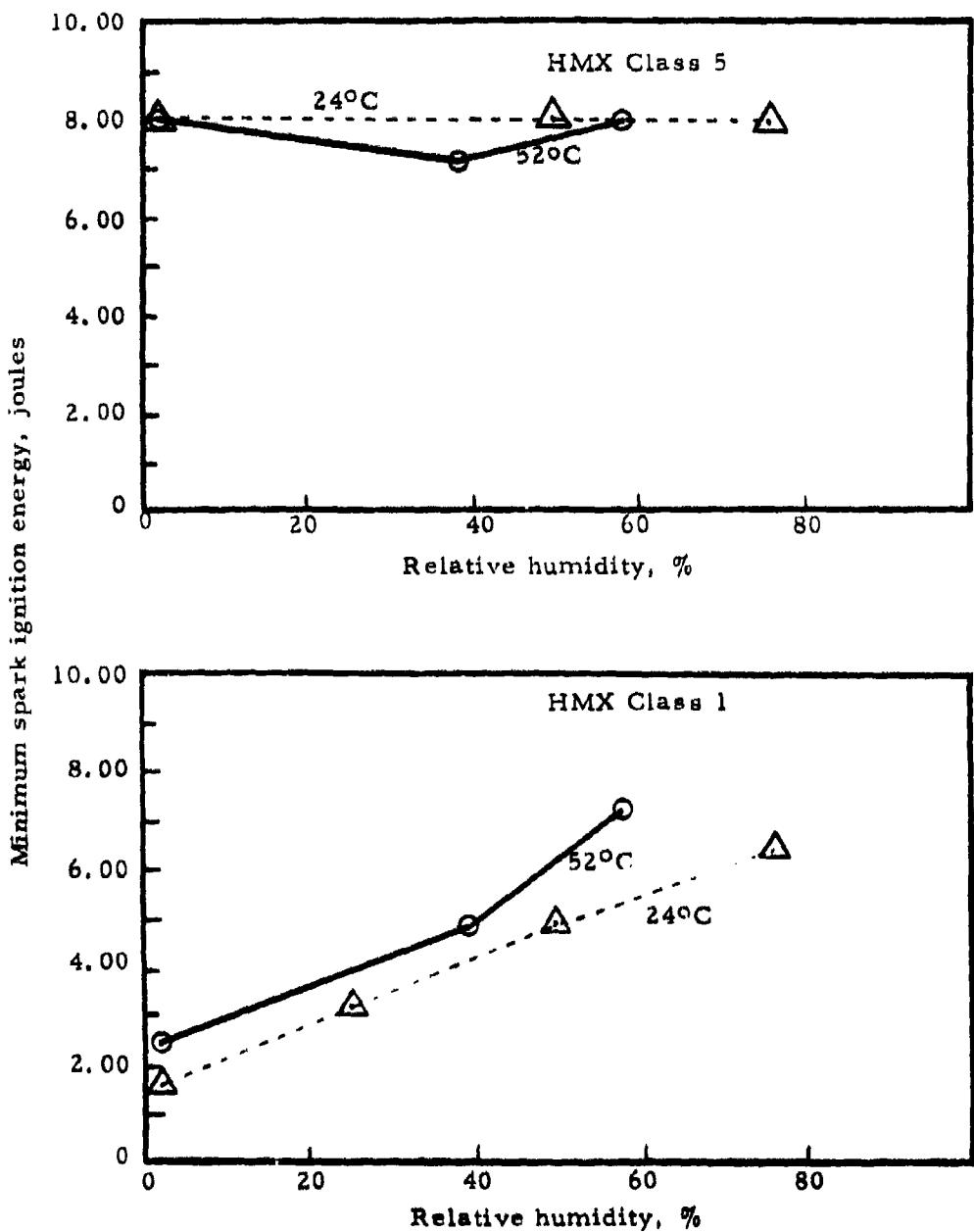


Figure 19. Minimum spark ignition energy versus relative humidity, HMX explosive, particle size class 5 and class 1.

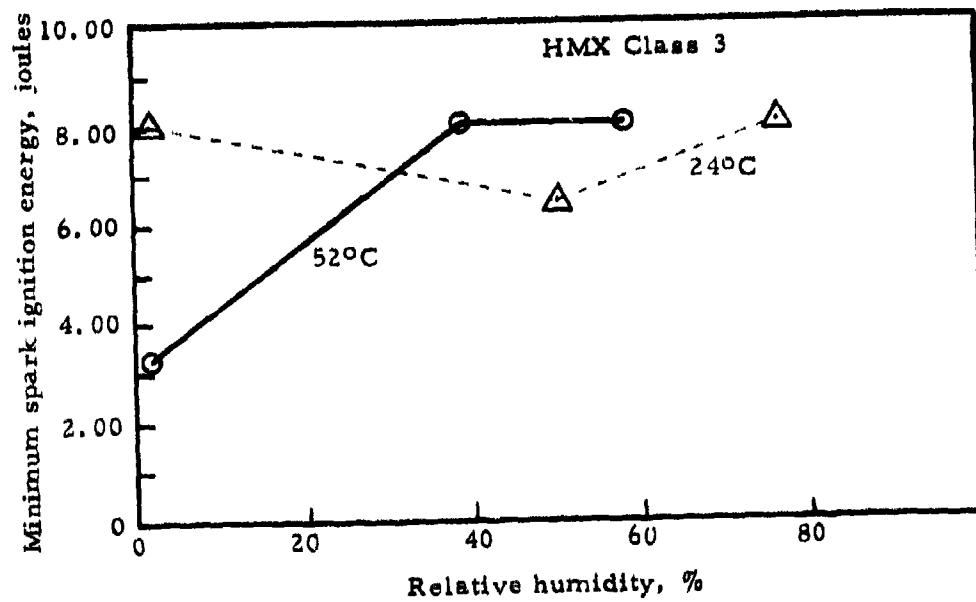


Figure 20. Minimum spark ignition energy versus relative humidity, HMX explosive, particle size class 3.

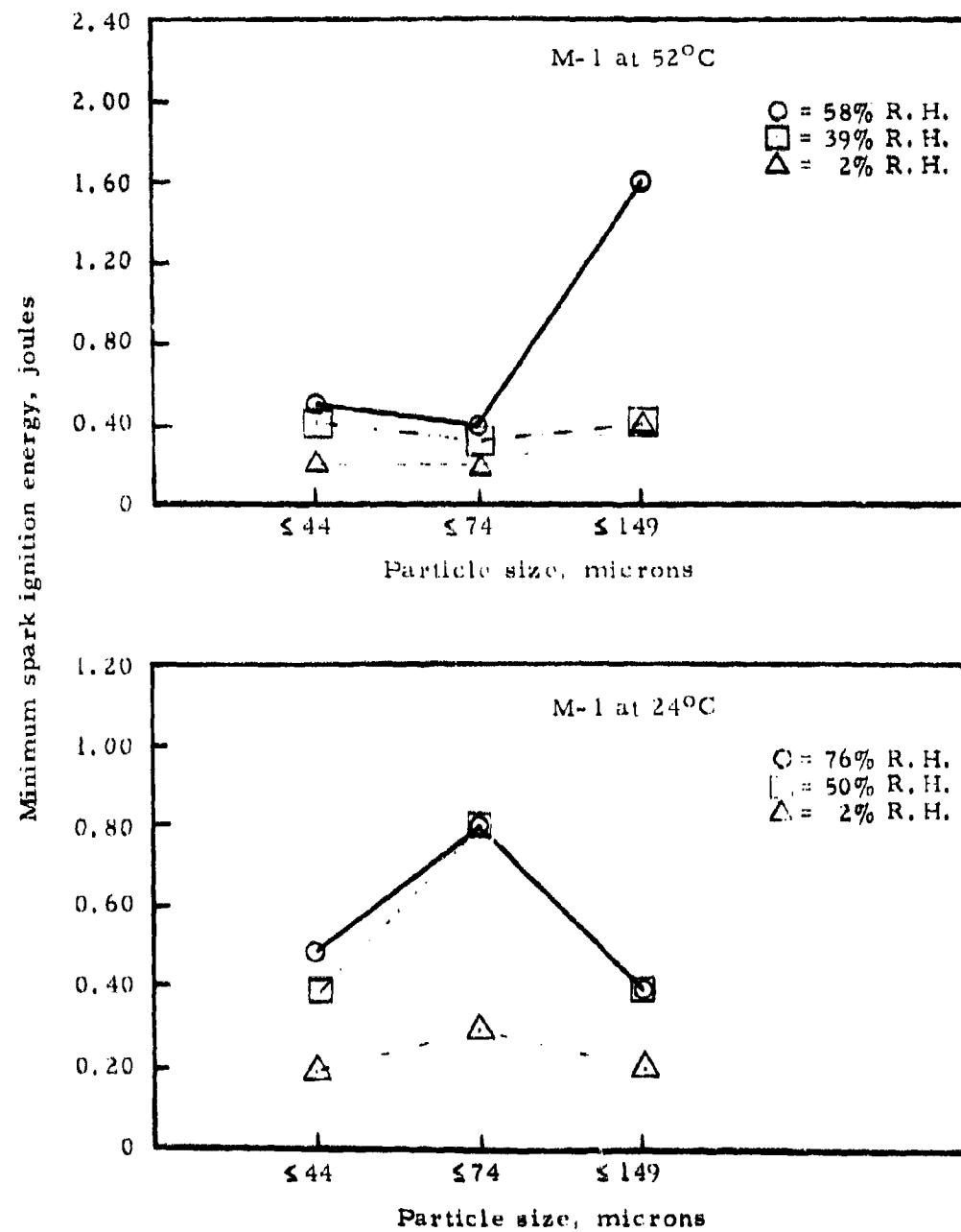


Figure 21. Minimum spark ignition energy versus particle size, M-1 propellant.

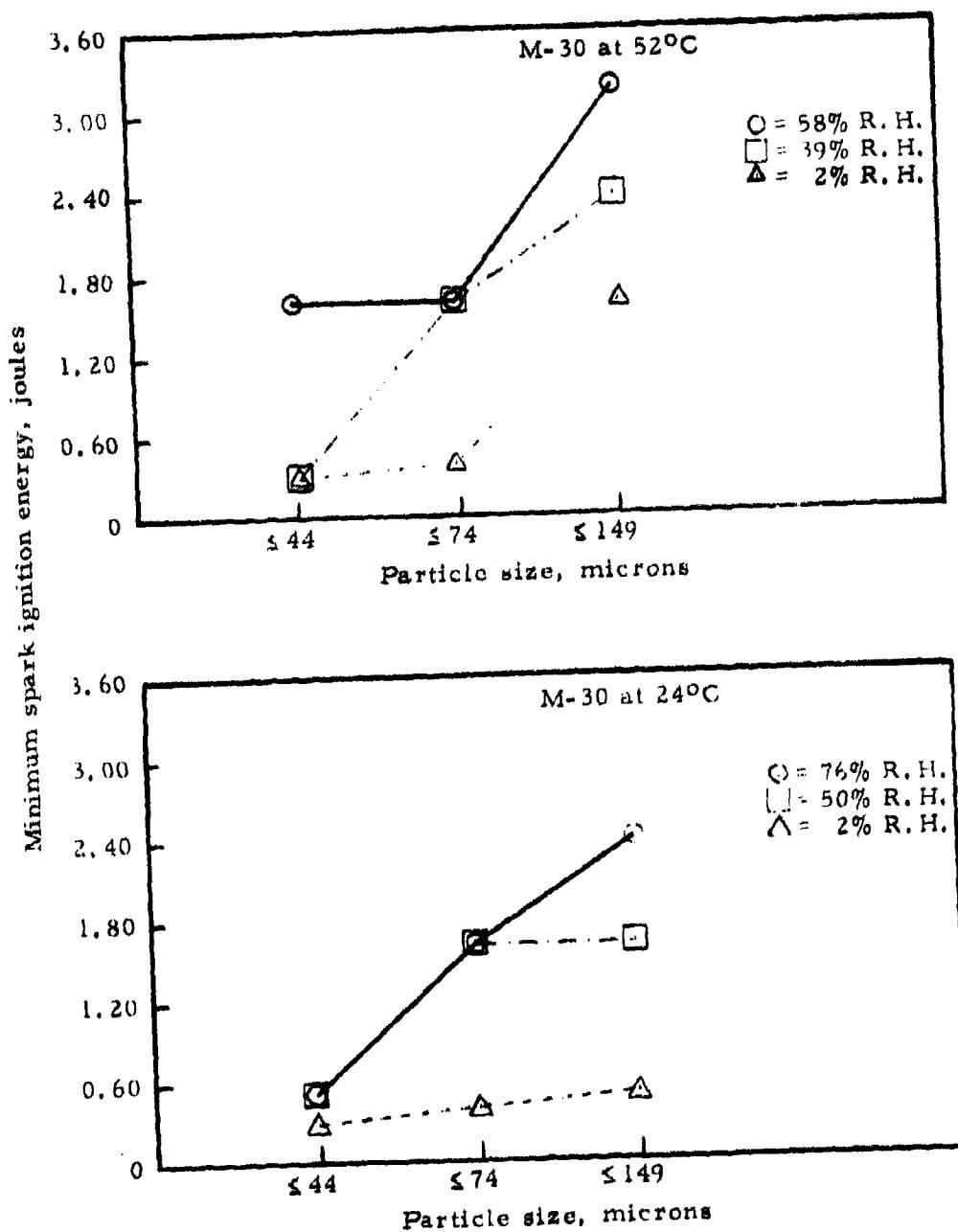


Figure 22. Minimum spark ignition energy versus particle size, M-30 propellant.

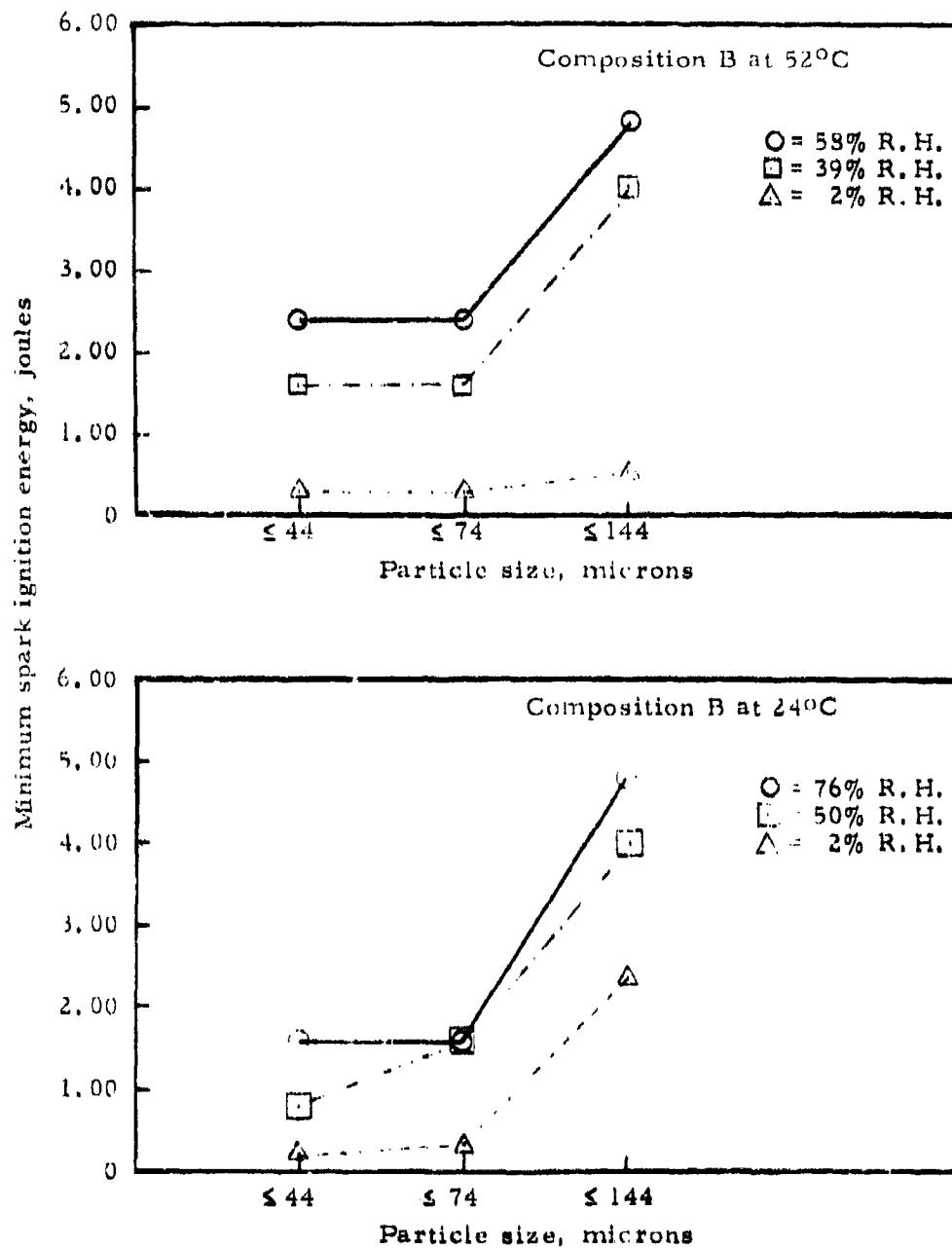


Figure 23. Minimum spark ignition energy versus particle size, Composition B explosive.

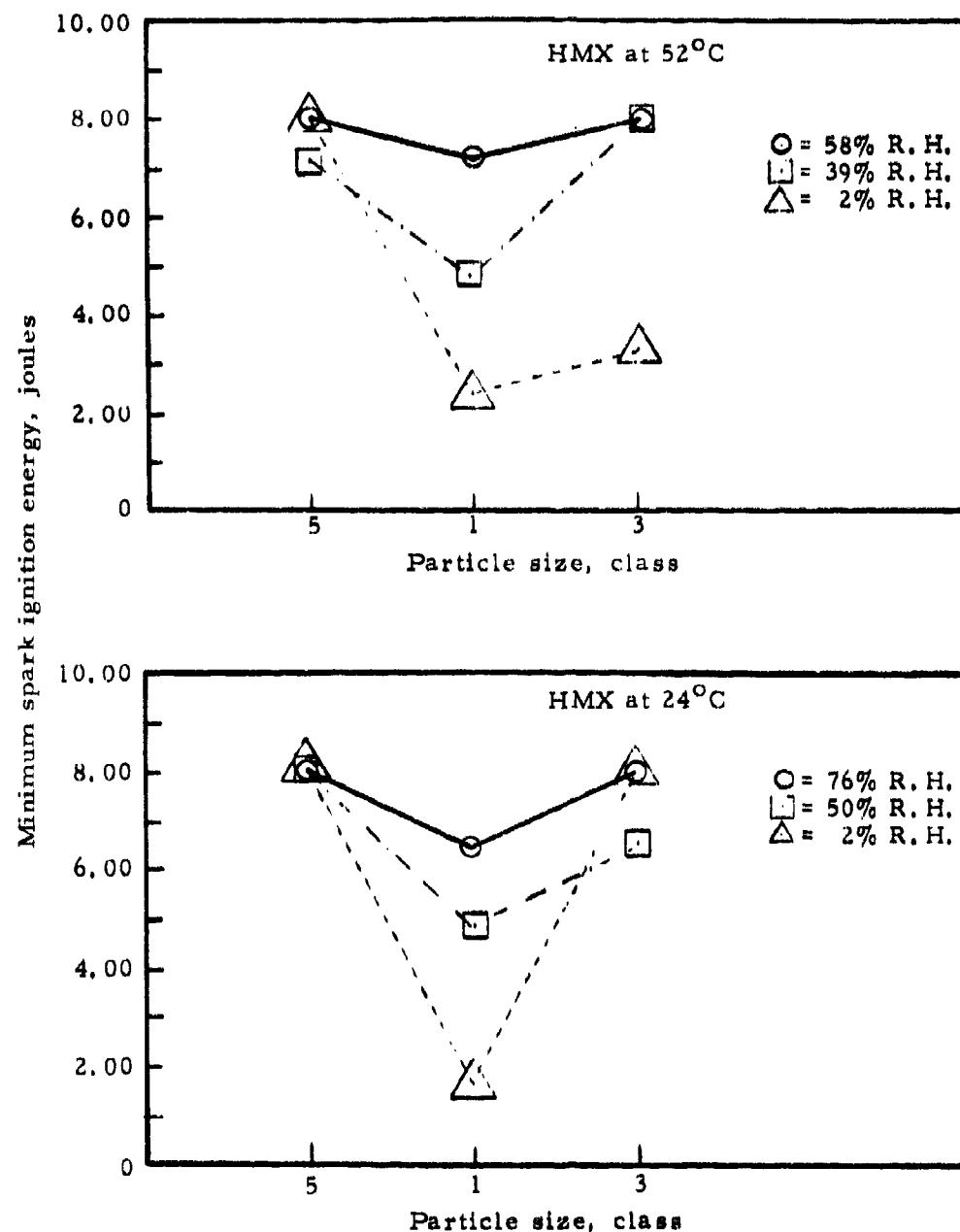


Figure 24. Minimum spark ignition energy versus particle size, HMX explosive.

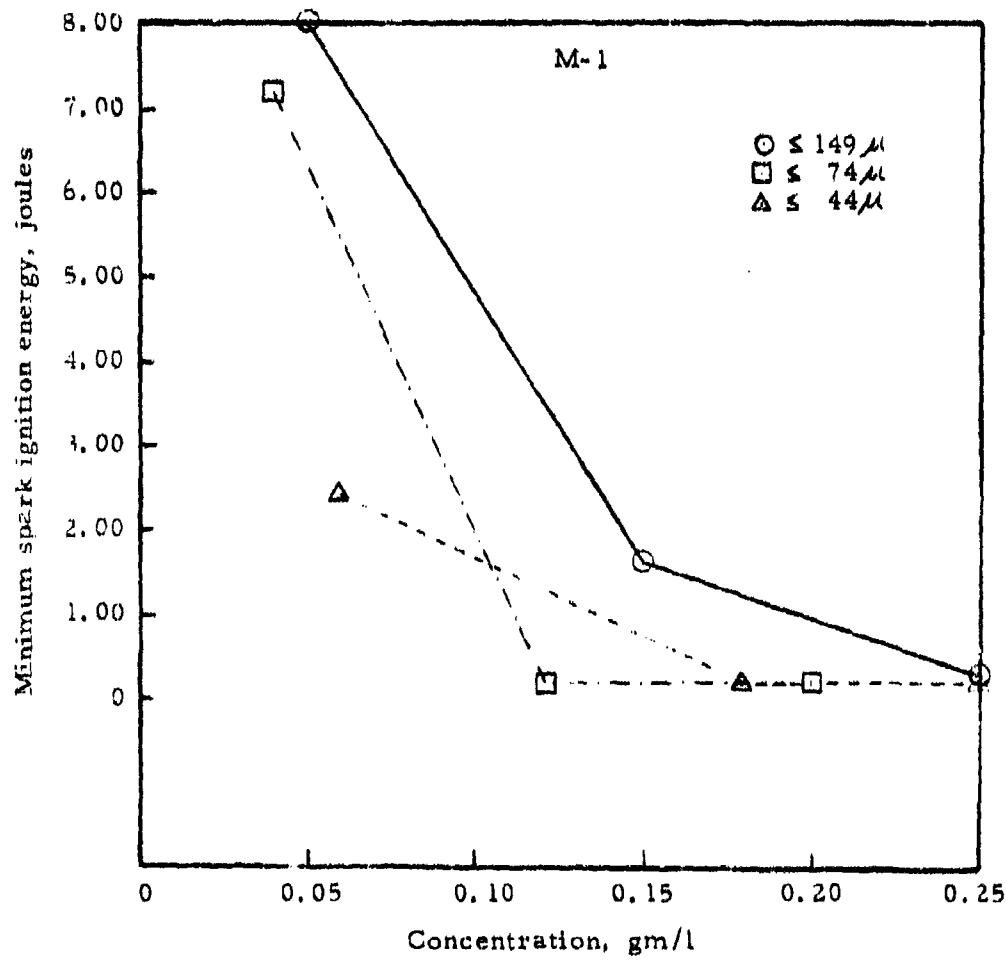


Figure 25. Minimum spark ignition energy versus concentration, M-1 propellant.

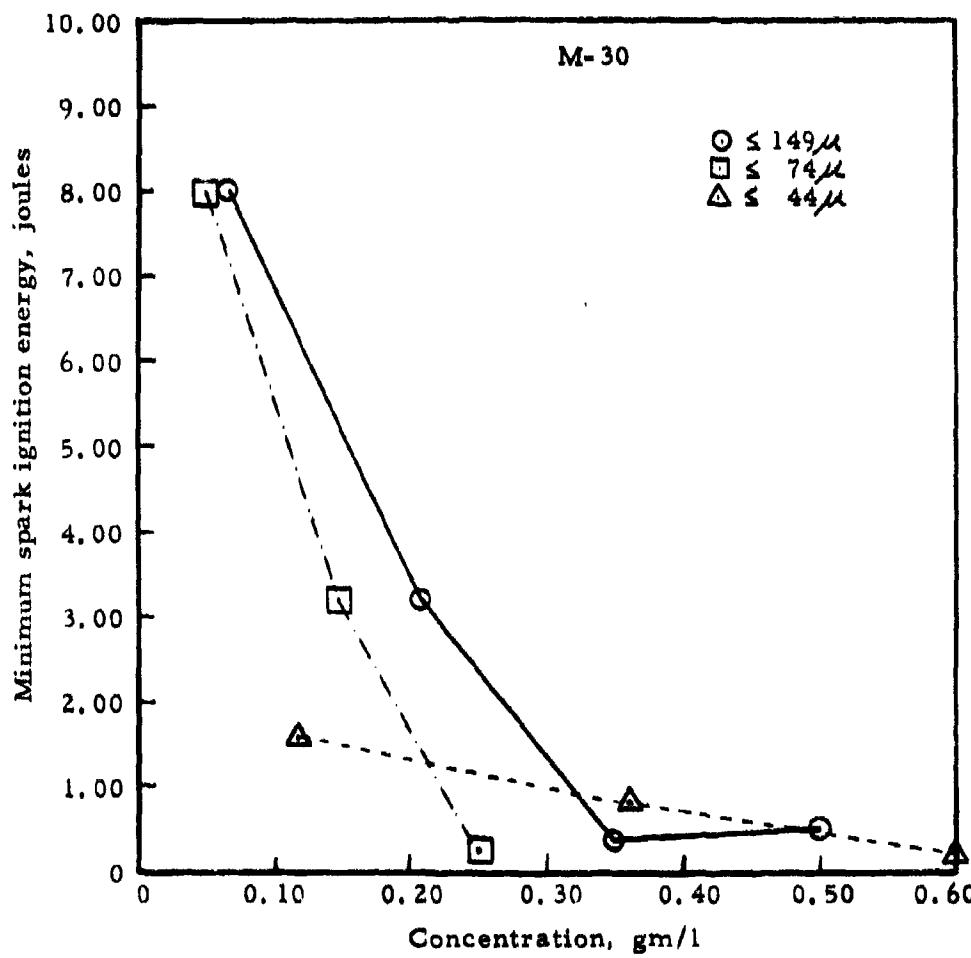


Figure 26. Minimum spark ignition energy versus concentration, M- 30 propellant.

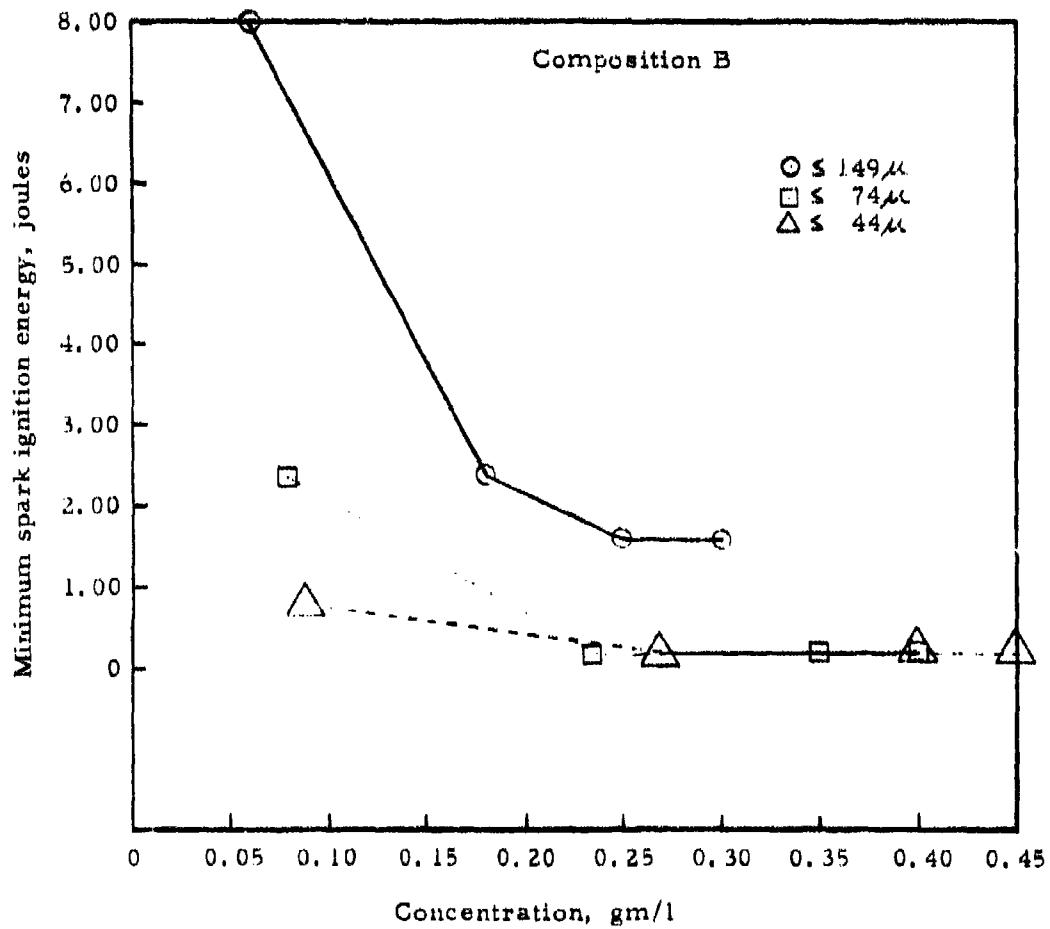


Figure 27. Minimum spark ignition energy versus concentration, Composition B explosive.

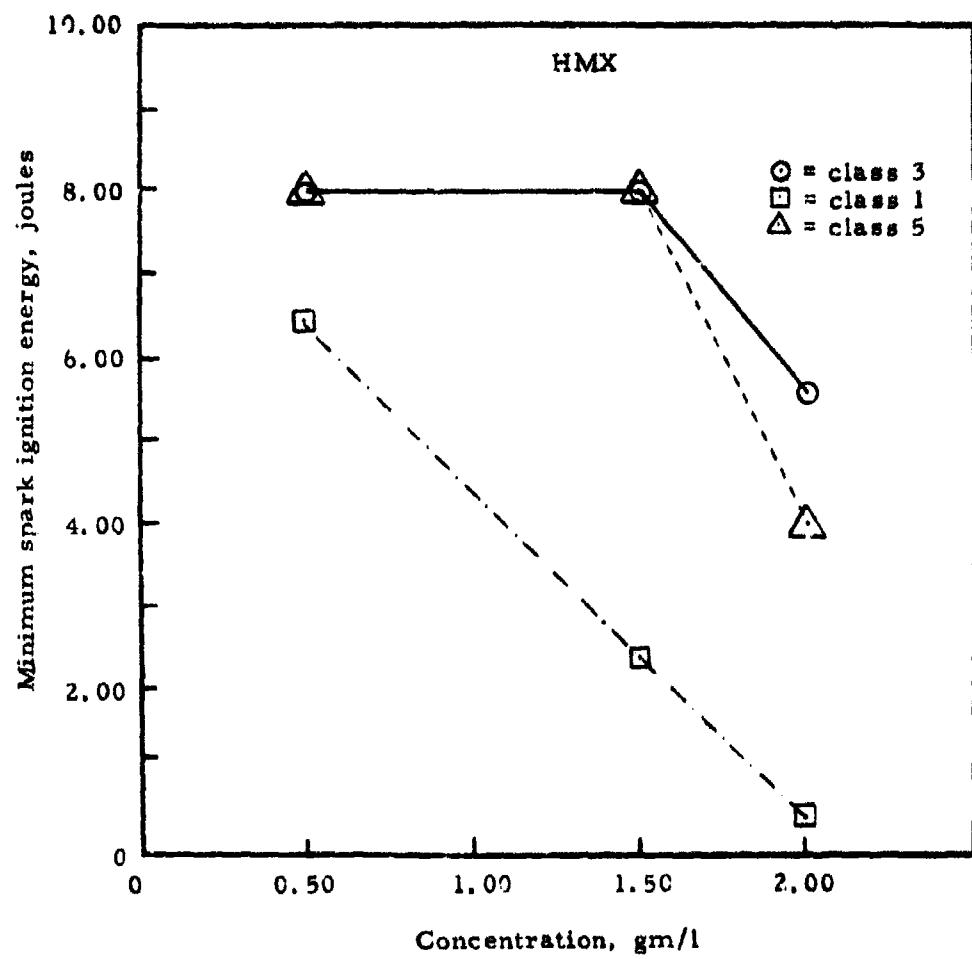


Figure 28. Minimum spark ignition energy versus concentration, HMX explosive.

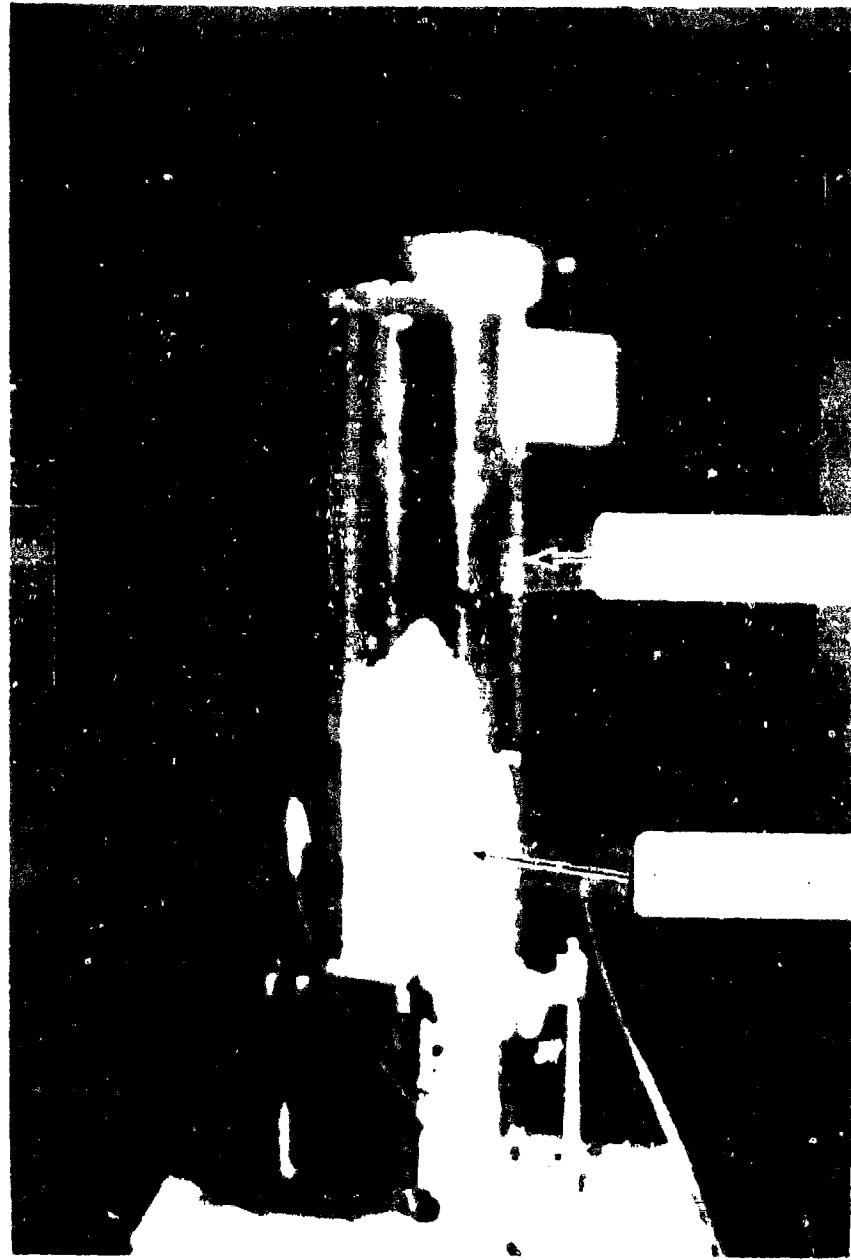


FIGURE 29. EARLY STAGE OF M-1 DUST RISING IN TEST CHAMBER.

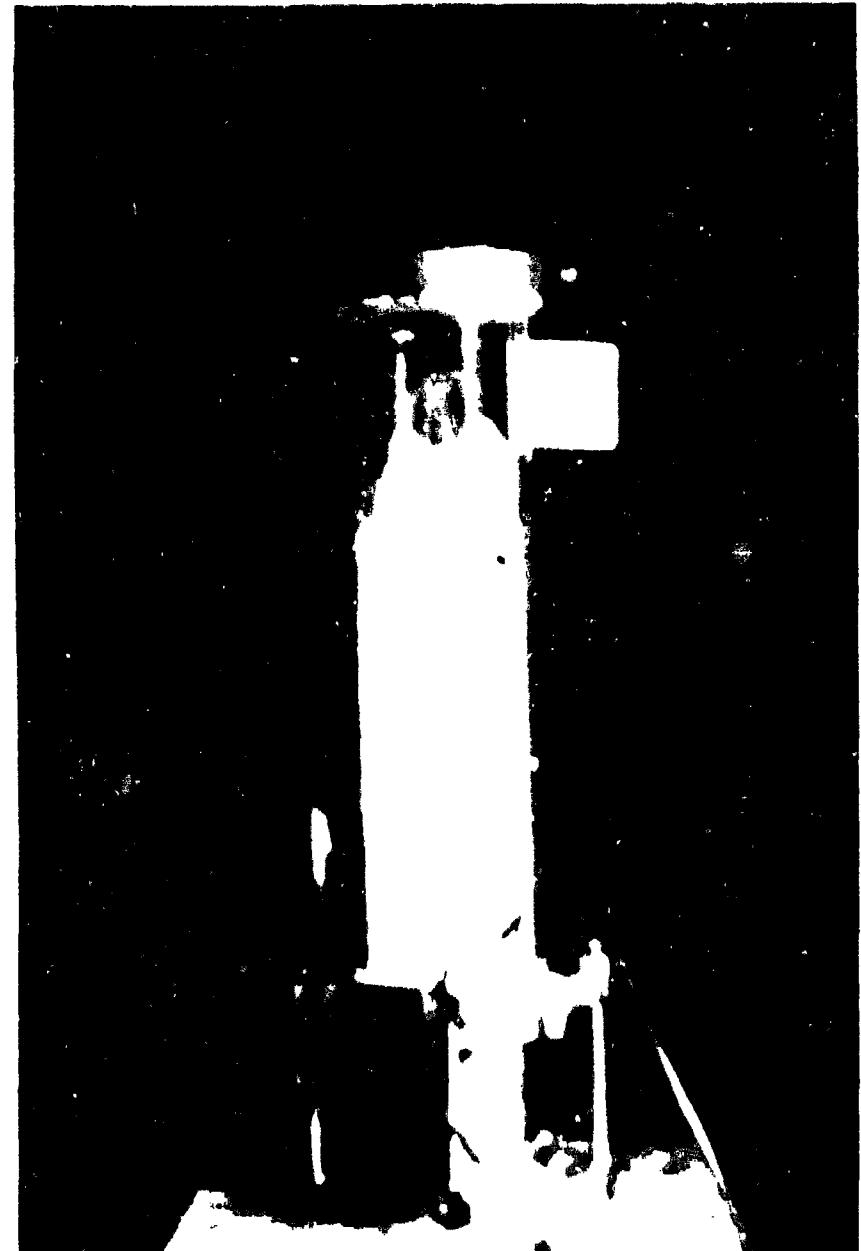


FIGURE 30. M-1 DUST RISING IN CHAMBER, 0.1 SEC AFTER RELEASE.

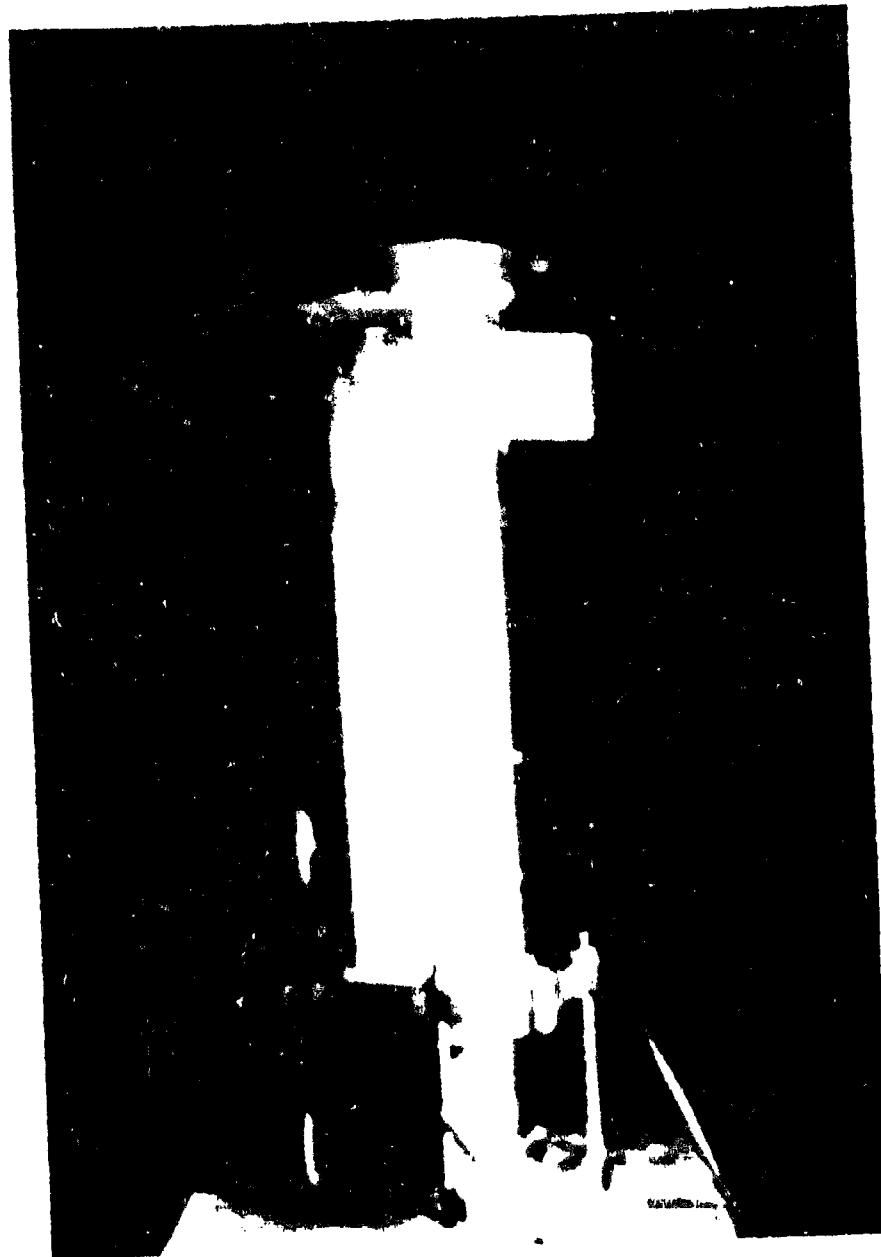


FIGURE 31. M-1 DUST RISING IN CHAMBER, 0.2 SEC AFTER RELEASE.

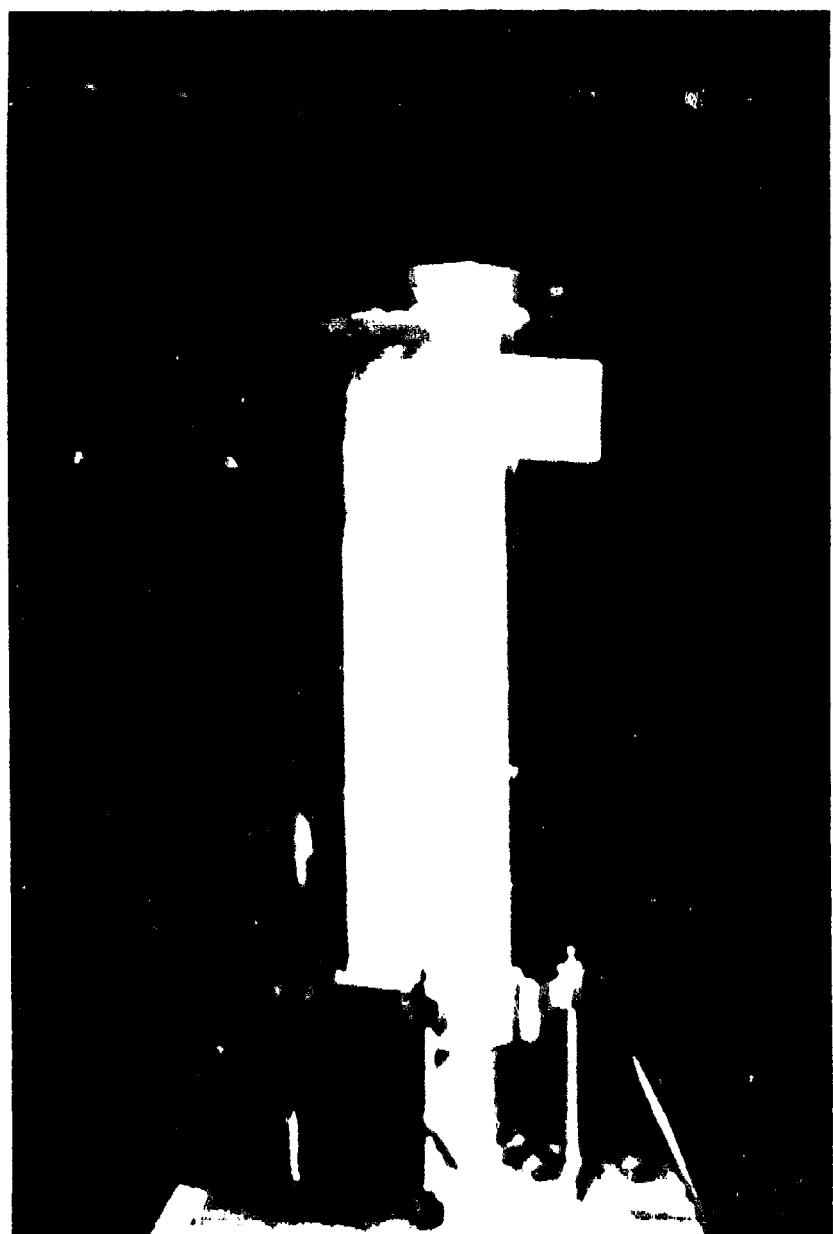


FIGURE 32. M-1 DUST DISPERSED IN CHAMBER JUST PRIOR TO SPARK IGNITION (0.5 SEC).

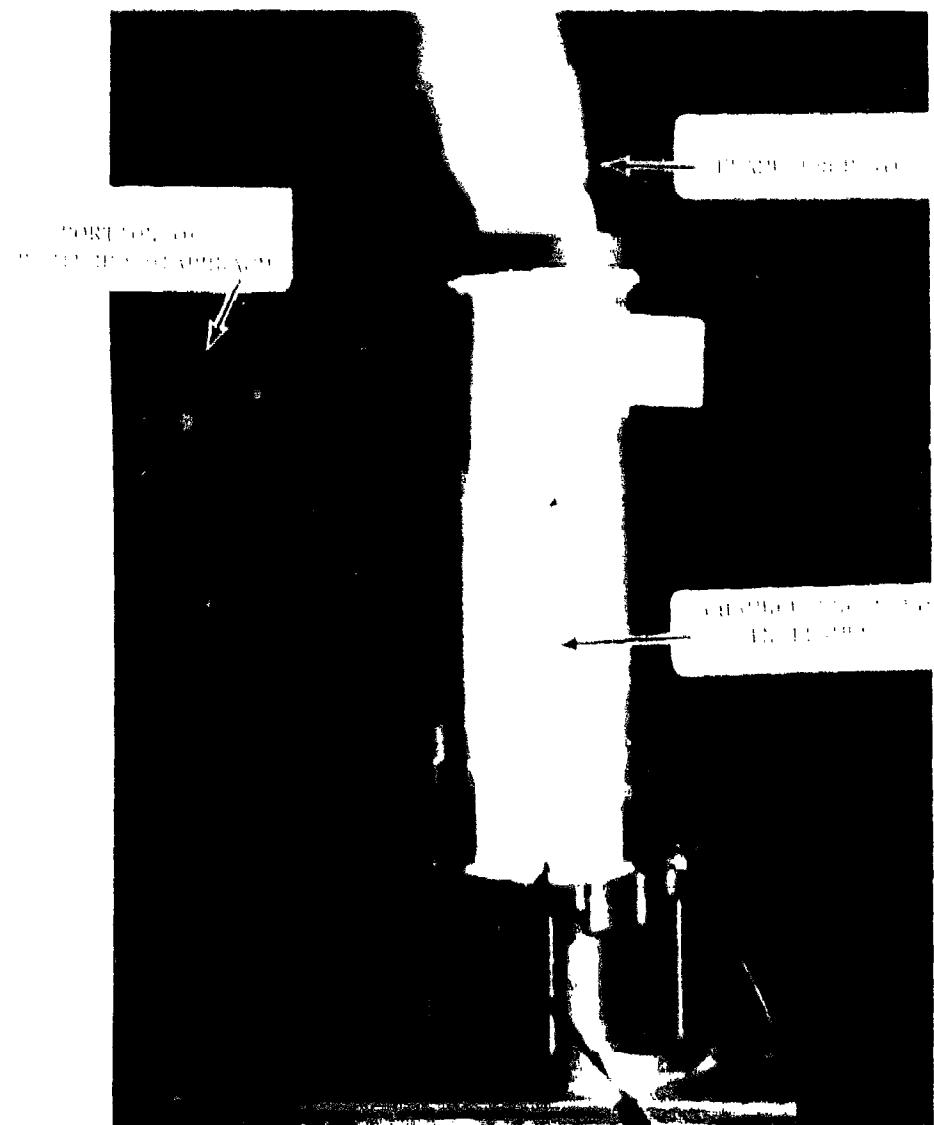


FIGURE 33. EXPLOSION OF DUST, RUPTURE OF DIAPHRAGM, AND FLAME ERUPTION, 0.1 SEC. AFTER SPARK IGNITION.

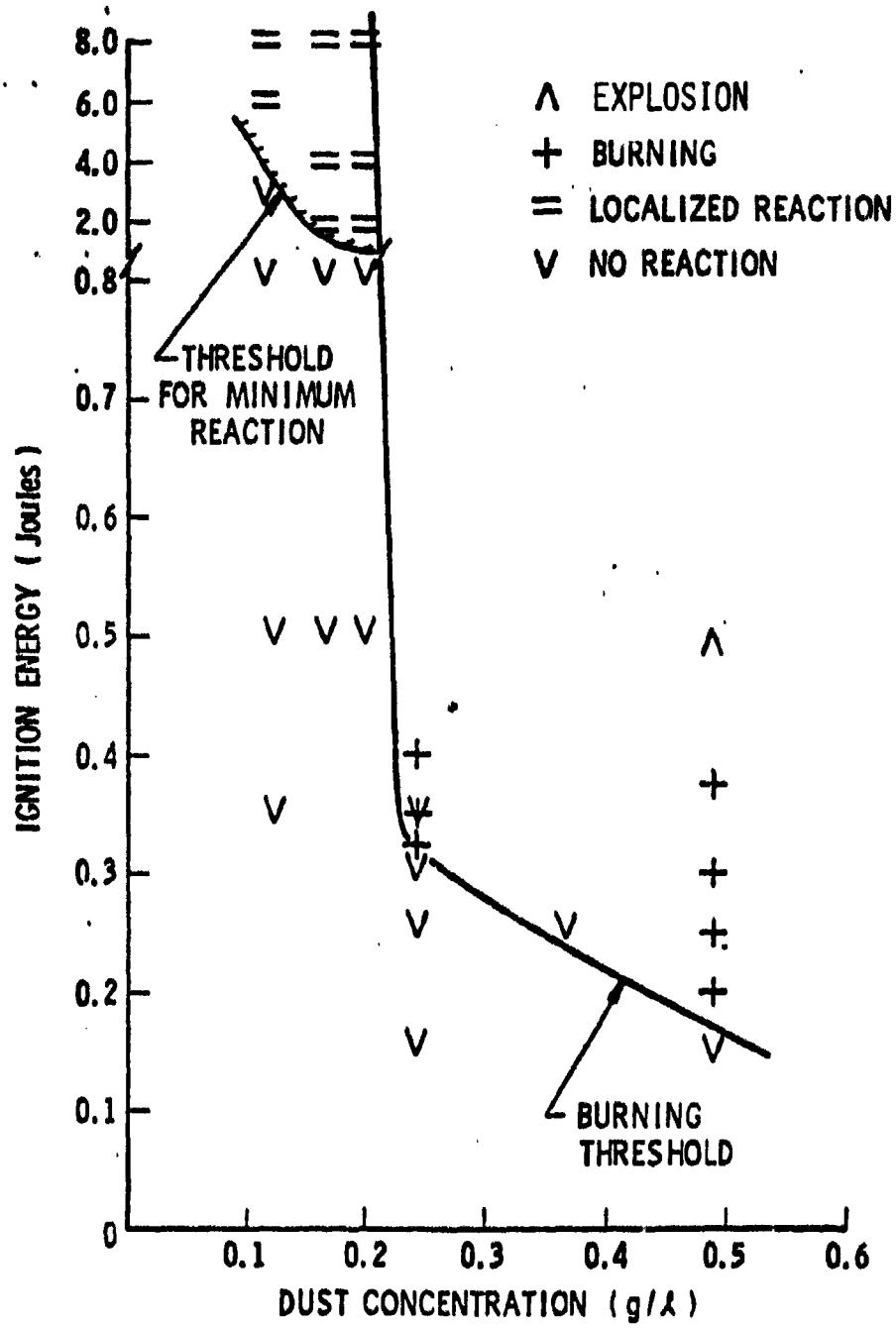


FIGURE 34. Dry M-1 Propellant Dust Explosibility
(Thru No. 200 Sieve, ≤ 75 Microns).

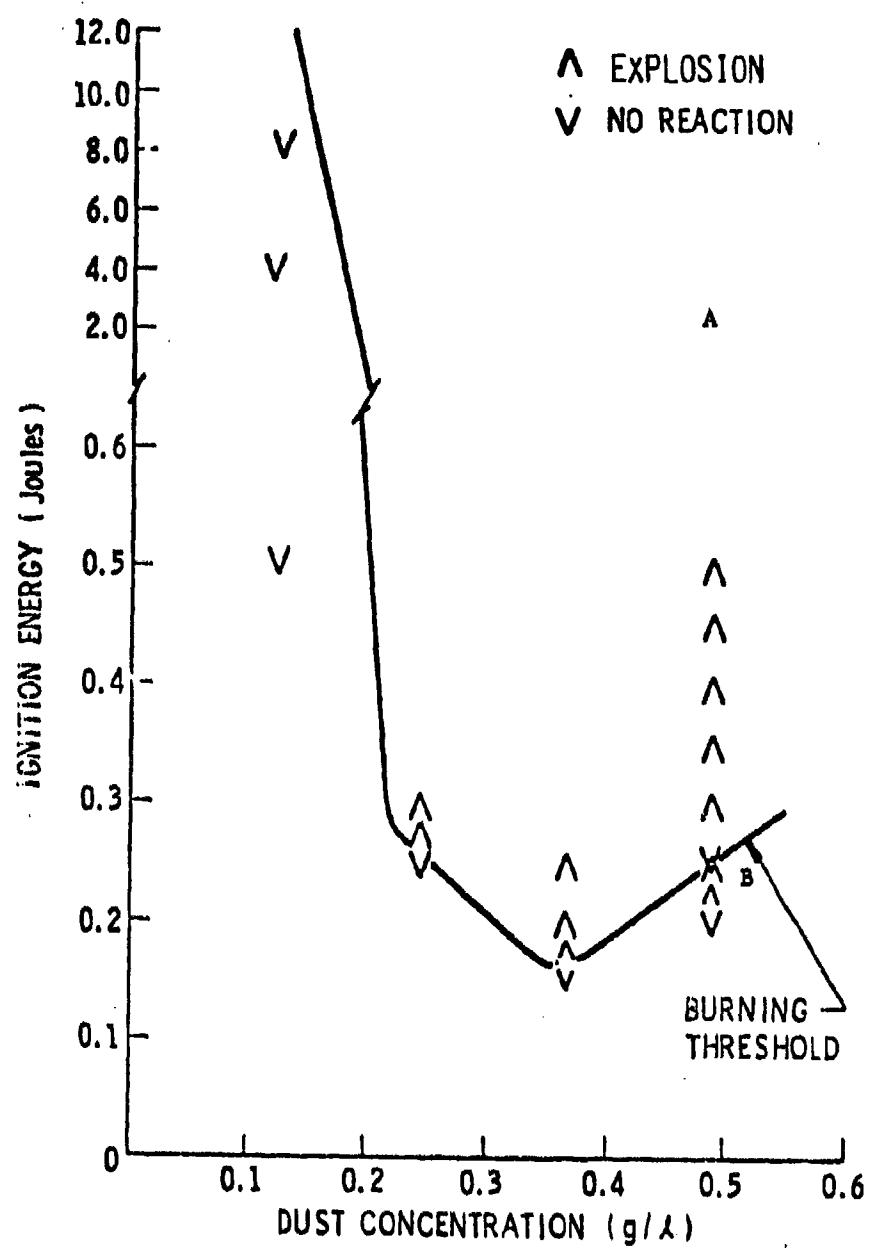


FIGURE 35. Dry M-1 Propellant Dust Explosibility
(Particle Size Between 75 and 105 Microns).

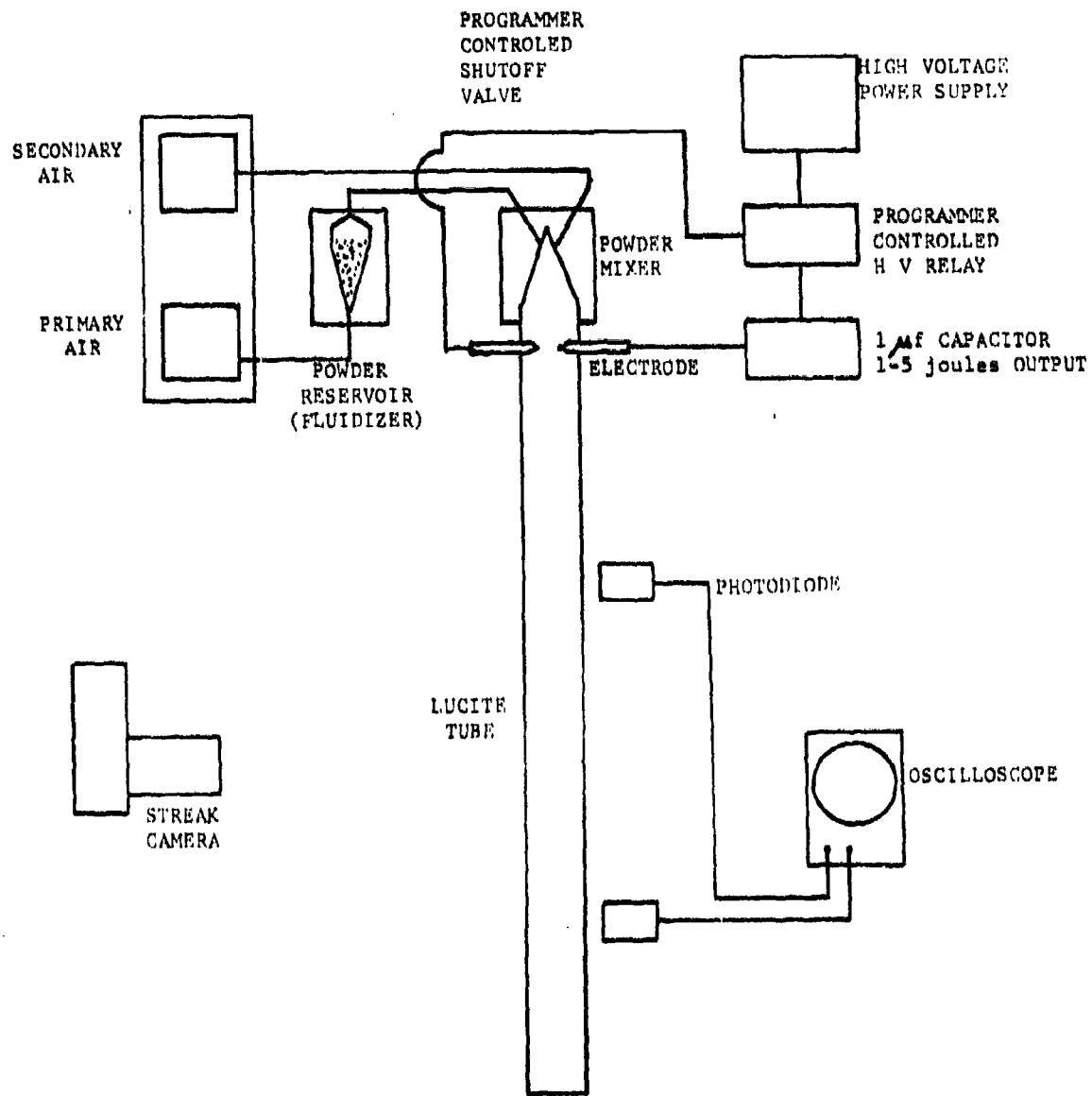


FIG 36. SCHEMATIC OF SPARK IGNITION TEST APPARATUS.

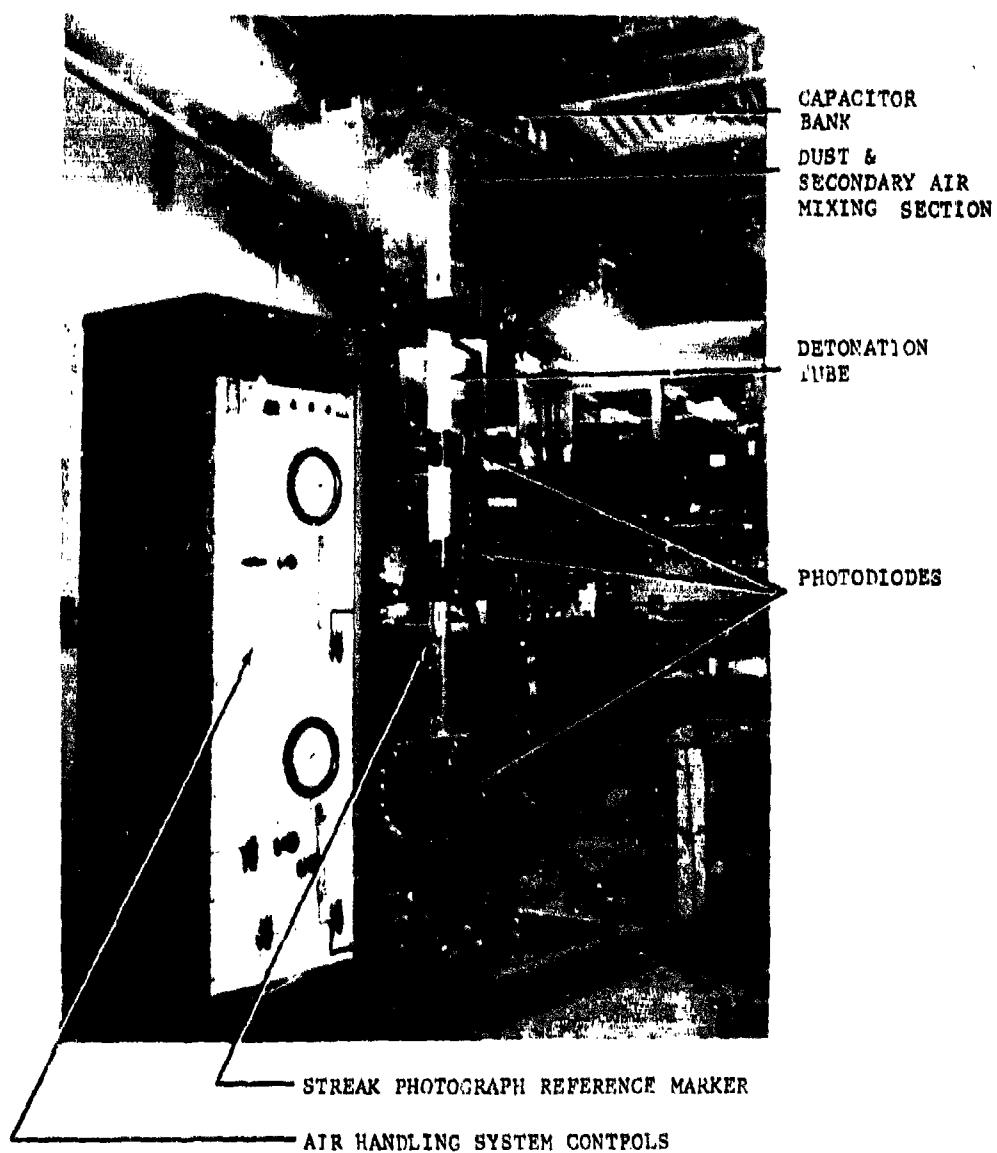
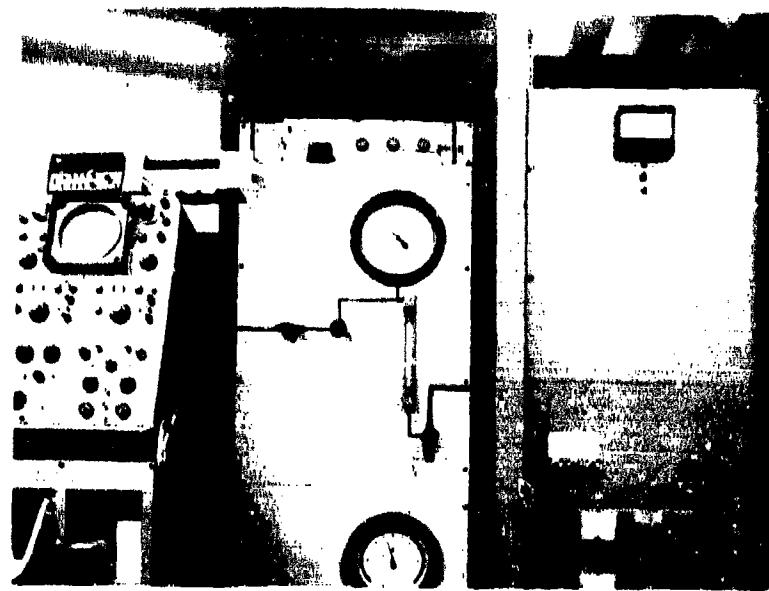


FIG 57. SPARK IGNITION TEST APPARATUS.



OSCILLOSCOPE

AIR HANDLING
SYSTEM CONTROL

HIGH VOLTAGE
POWER SUPPLY

FIG 3B. SPARK IGNITION TEST APPARATUS CONTROL PANEL.

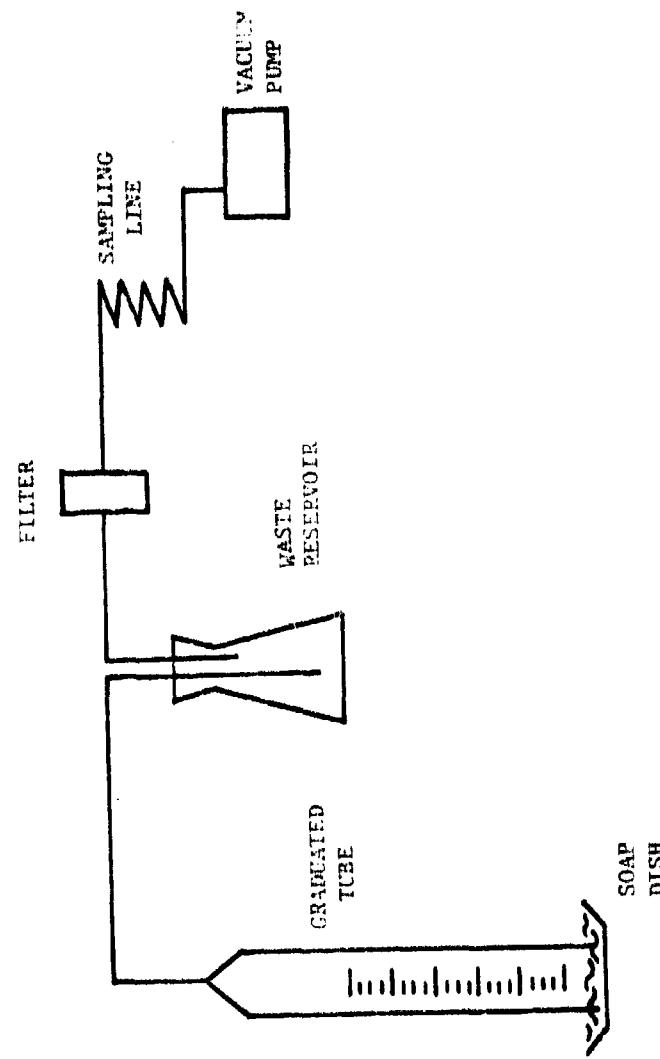


FIG 39. CALIBRATING SAMPLING PUMP APPARATUS.

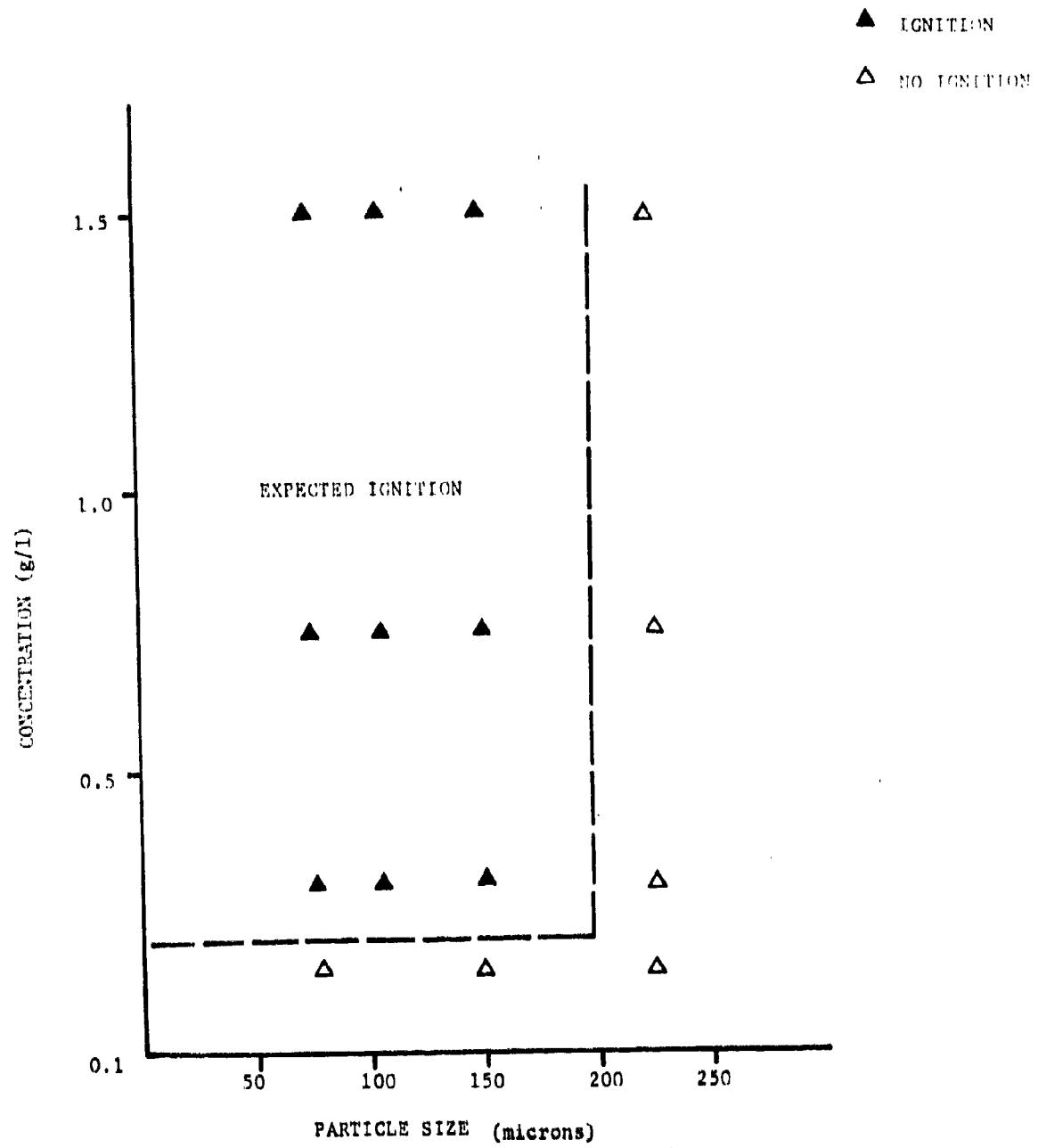


FIG 40. EXPLOSIVE DUST CONCENTRATION VERSUS PARTICLE SIZE FOR HMX.

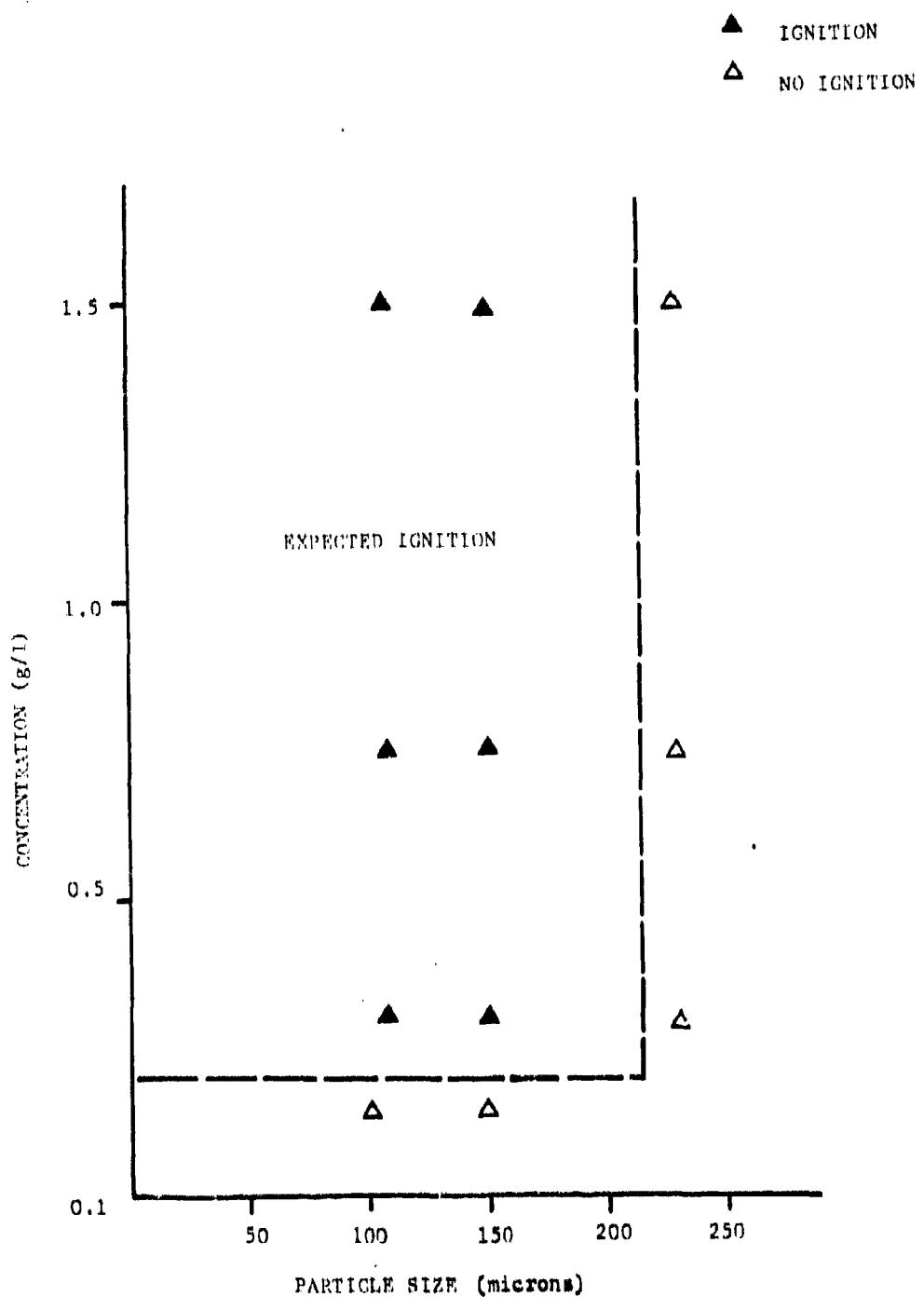


FIG 41. EXPLOSIVE DUST CONCENTRATION VERSUS PARTICLE SIZE FOR RDX.

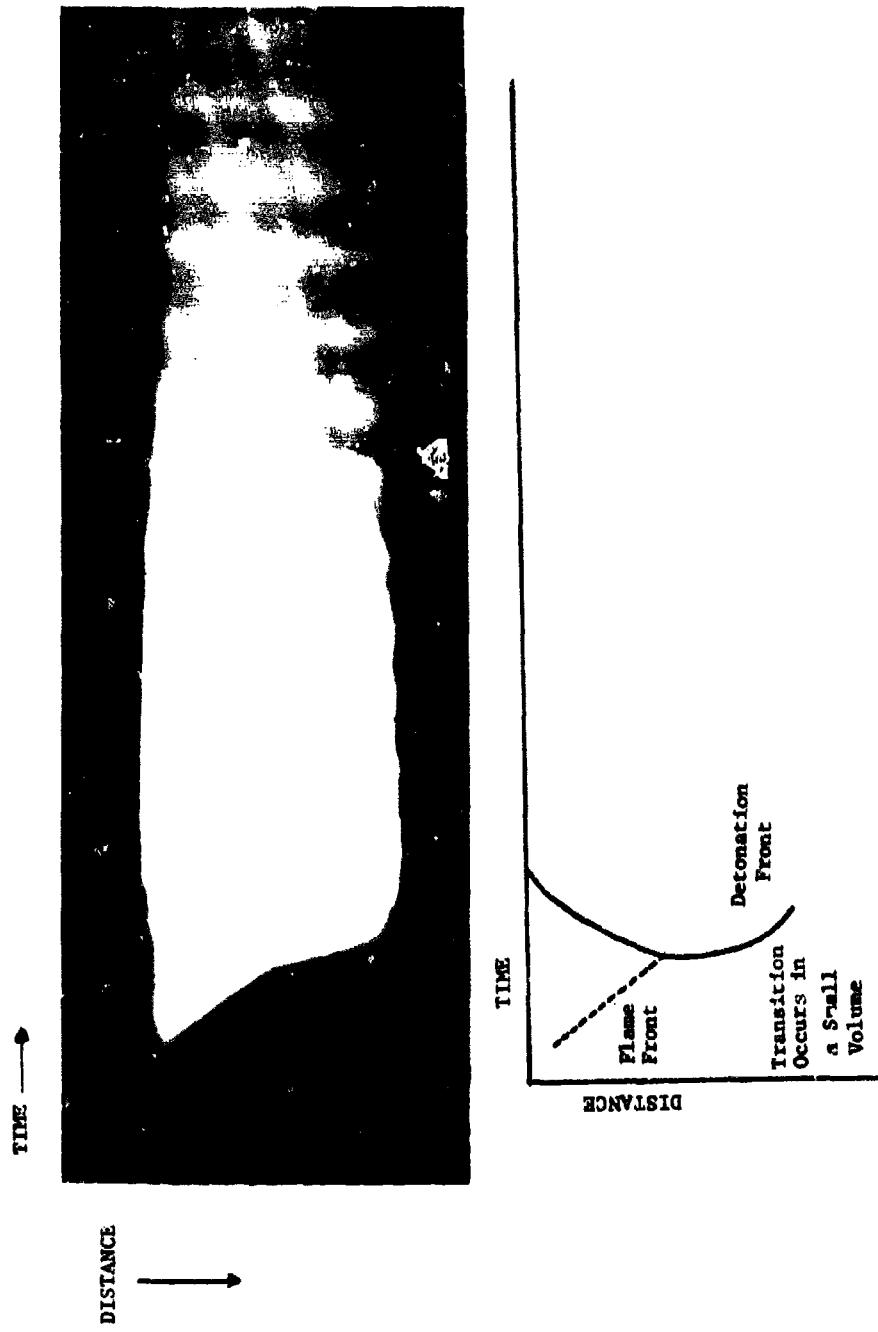


FIG. 42. DEFLAGRATION TO DETONATION TRANSITION FOR A SPARK INITIATED STOICHIOMETRIC PROPANE-OXYGEN MIXTURE.

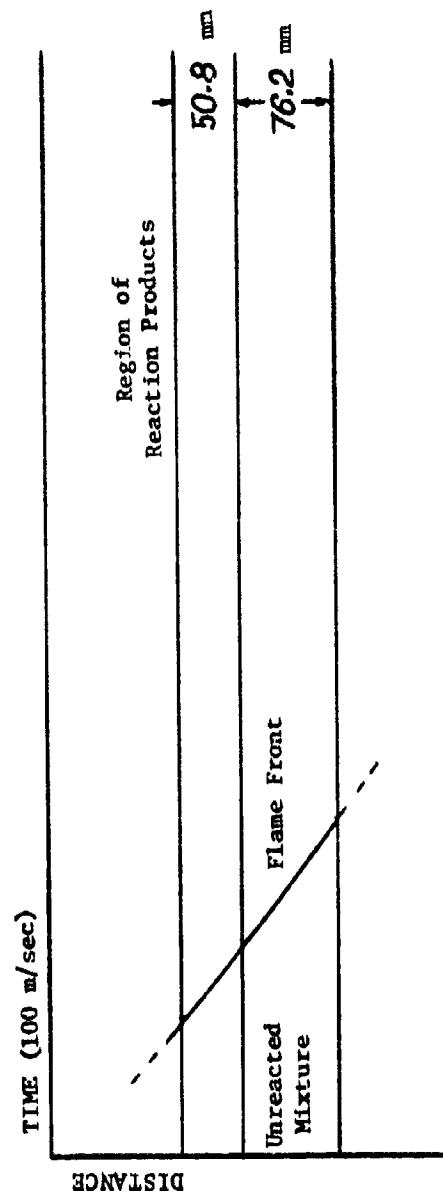
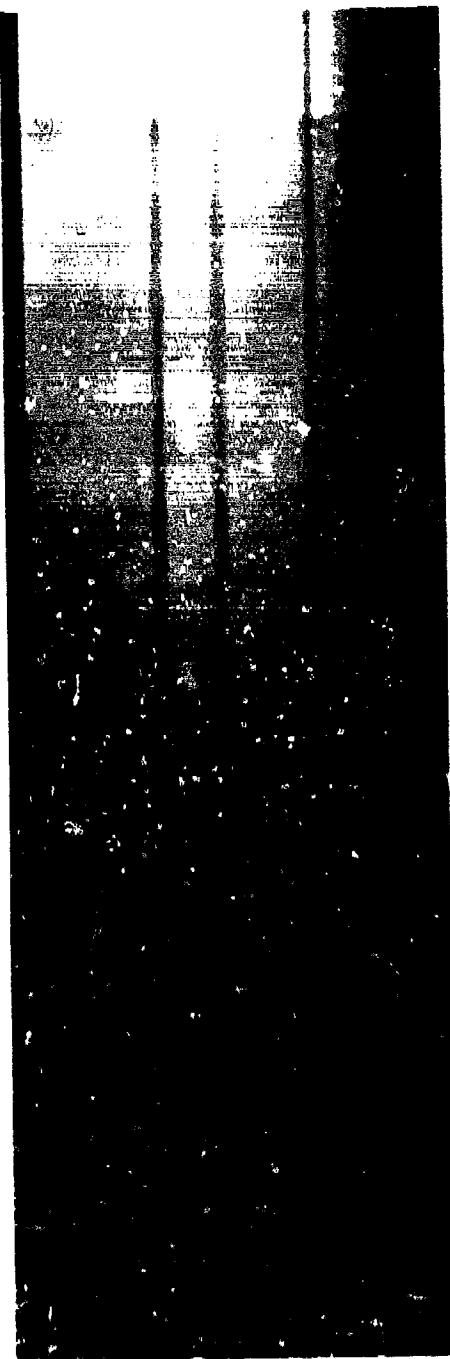


FIG 43. FLAME PROPAGATION THROUGH SAMPLES OF RDX POWDER IN AIR.

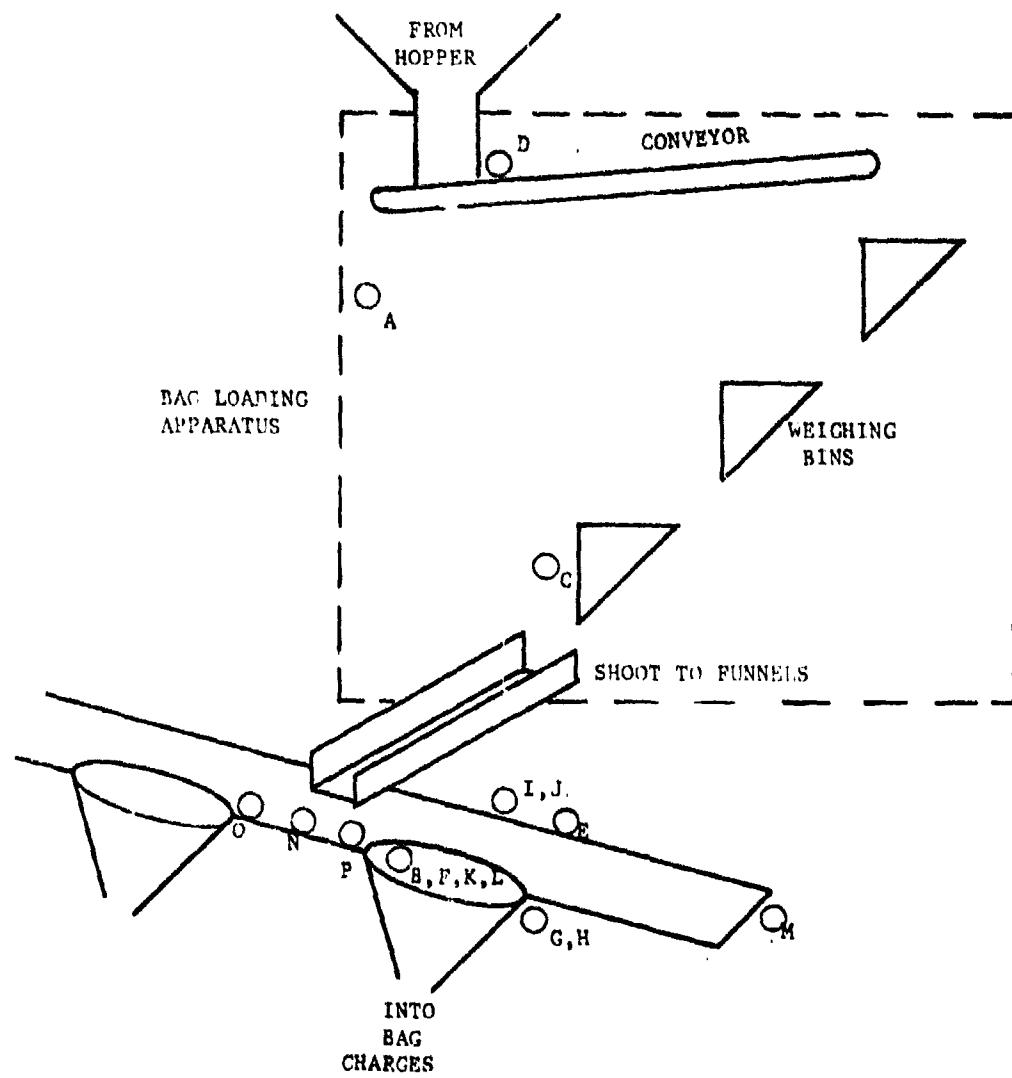


FIG 44 LOCATIONS OF SAMPLING SITES AT INDIANA ARMY AMMUNITION PLANT LOADING OPERATION

APPENDIX A. THE HARTMANN APPARATUS

Two of the greatest difficulties in dust explosibility tests are production and maintaining a uniform dust cloud. In testing, it is desirable to have as small a dust cloud as possible to avoid dangerous, powerful explosions. Yet, confining a dust cloud to a small volume poses some problems. The dispersed dust particles will tend to rapidly adhere to the sides of the container. Also, since the dust particles fall at a rate dependent on size, the particles must either be replenished, or the dust cloud must be ignited before the concentration has decreased from particles falling or adhering to the container walls. There are some devices which, through a metering out of dust particles, attempt to replenish those lost from the dust cloud. The Hartmann Apparatus, on the other hand, produces a momentary dust cloud from a blast of air which passes through a dust sample. After a short period of turbulence, a momentarily uniform dust cloud is produced, at which time, an attempt should be made to ignite the dispersed dust. The Hartmann Apparatus can be used in all of the dust explosibility tests.

The principal components of the Hartmann Apparatus (see Figures A1, A2, A3) consist of a 30.5-cm vertically mounted Lucite tube, and the dispersion cup to which the Lucite tube is attached. The combined volume of the Lucite tube and the dispersion cup (i.e., the assumed volume of the dust cloud) is 1.23 liters. This is the combustion chamber for the dust. Components can be divided into two groups: the dust dispersion elements and the ignition elements. A weighed amount of dust is placed in the dispersion cup around a mushroom shaped deflector in the bottom of the dispersion cup. The deflector serves two purposes: it deflects the incoming air so as to disperse the dust sample, and it regulates the rate at which air can flow from the compressed air reservoir into the Lucite tube. The compressed air reservoir, with a volume of 1.31 liters, is used to produce a blast of air to disperse the dust lying in the bottom of the dispersion cup. The pressure used in the reservoir can be between 3.4×10^4 to 10.3×10^4 pascals (5 to 15 psi). However, 6.8×10^4 pascals (10 psi) were used for all tests conducted. The compressed air in the reservoir is released by means of a solenoid valve, and a check valve is located between the solenoid valve and the dispersion cup. Its purpose is to prevent any flow of combustion gases back into the solenoid valve or reservoir.

Supporting all the components listed so far is the aluminum base. The Lucite tube can be rapidly attached to the dispersion

cup for a test, or detached for cleaning and reloading with dust by means of the hinged bolts. At the top of the Lucite tube is placed a paper diaphragm, held in place by the locking ring. The paper diaphragm confines the dispersed dust particles within the tube before ignition, yet allows incoming air from the reservoir to escape. The diaphragm will burst if the burning dust produces a pressure of at least 2.1×10^5 pascals (535.8 psi).

The ignition elements consist of two groups (two electrodes per group) of 20-gauge tungsten electrodes and electrode holders. The upper group, 10.2 cm above the bottom of the Lucite tube, was used in all tests conducted.

After the blast of air from the reservoir, a certain period of time must pass for the dust concentration to become fairly uniform in the test chamber, before attempting to ignite the dust cloud. Also, if too much time should elapse, the dust will settle out on the walls and bottom of the Lucite tube. It can be seen that delay timing between release of the compressed air and the discharge of the capacitors is essential. The timing is accomplished electronically. Upon initiating the solenoid valve by pushing a button, a timing sequence is initiated, which will trigger the capacitors to discharge into the luminous tube transformer at a later instant. This time delay is varied until an optimum value is found; i.e., one that occurred when the dust cloud was most uniform. Figure A4 is a block diagram of the electronic instruments used, and Figure A5 shows the control console that houses the electronics necessary to provide the variable electrical discharge.

In summary, the production of uniform dust clouds is difficult. An approximation to a uniform dust cloud can be made with the Hartmann Apparatus, which was used for dust explosibility tests. It should be realized that the Hartmann Apparatus will allow an accurate determination of relative explosibility of different dusts, but that the data obtained cannot always be directly applied to real life situations because of the empirical nature of the testing apparatus (40).

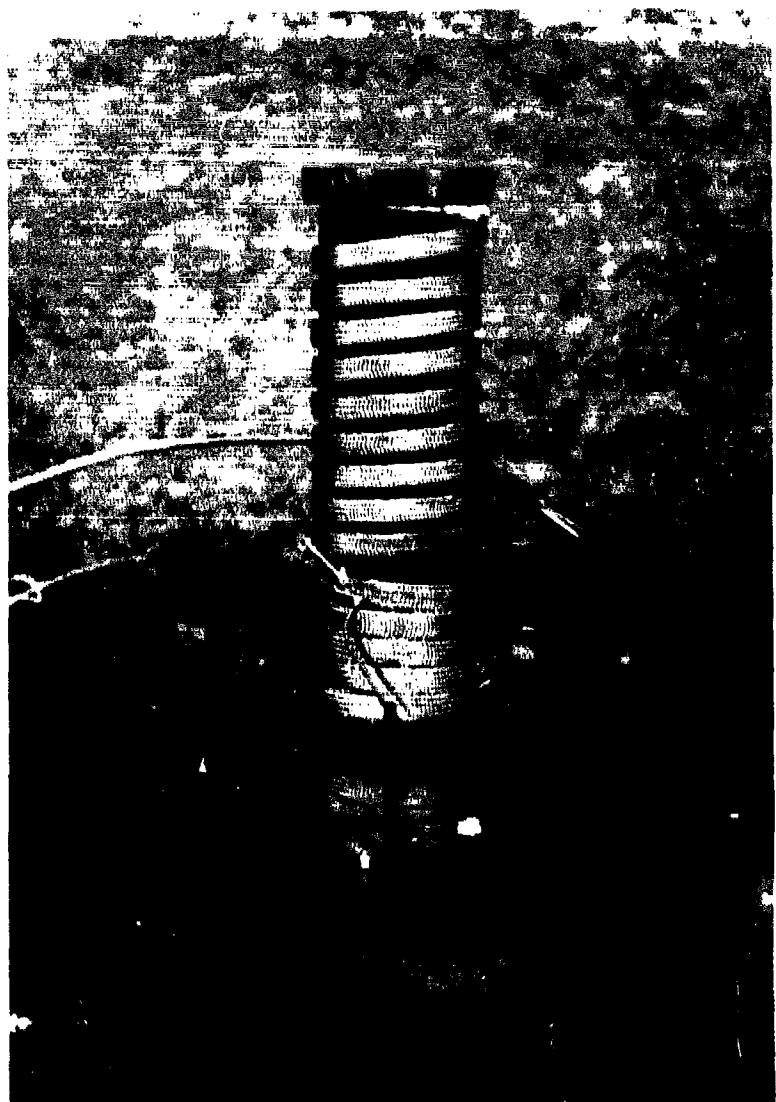


Figure A-1. Heated Hartmann Lucite tube
and dust dispersion cup.

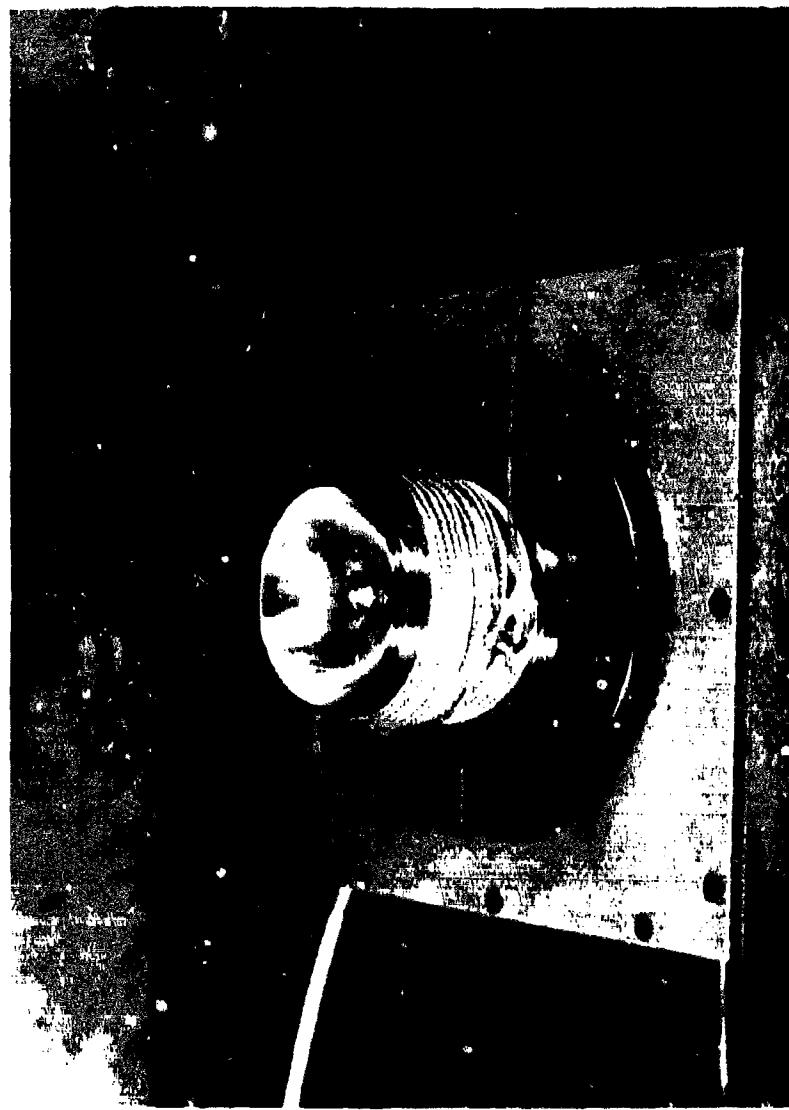


Figure A-2. Dust dispersion cup.

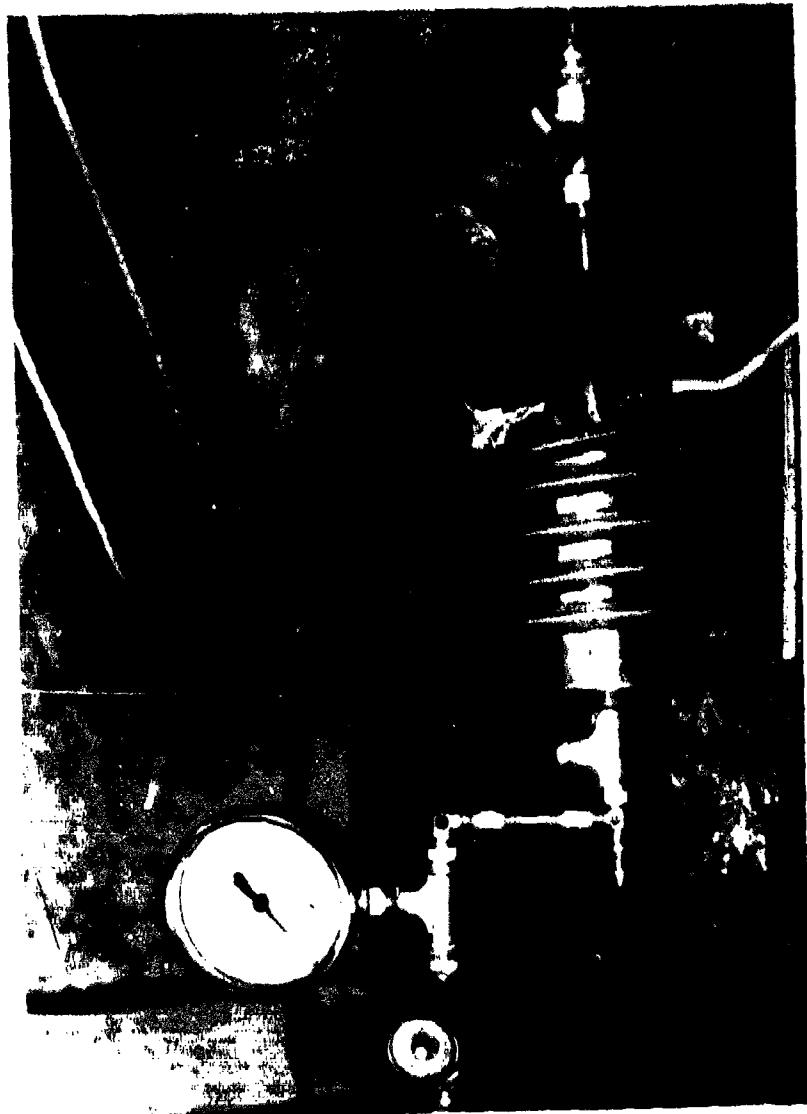


Figure A-3. Air reservoir for dust dispersion.

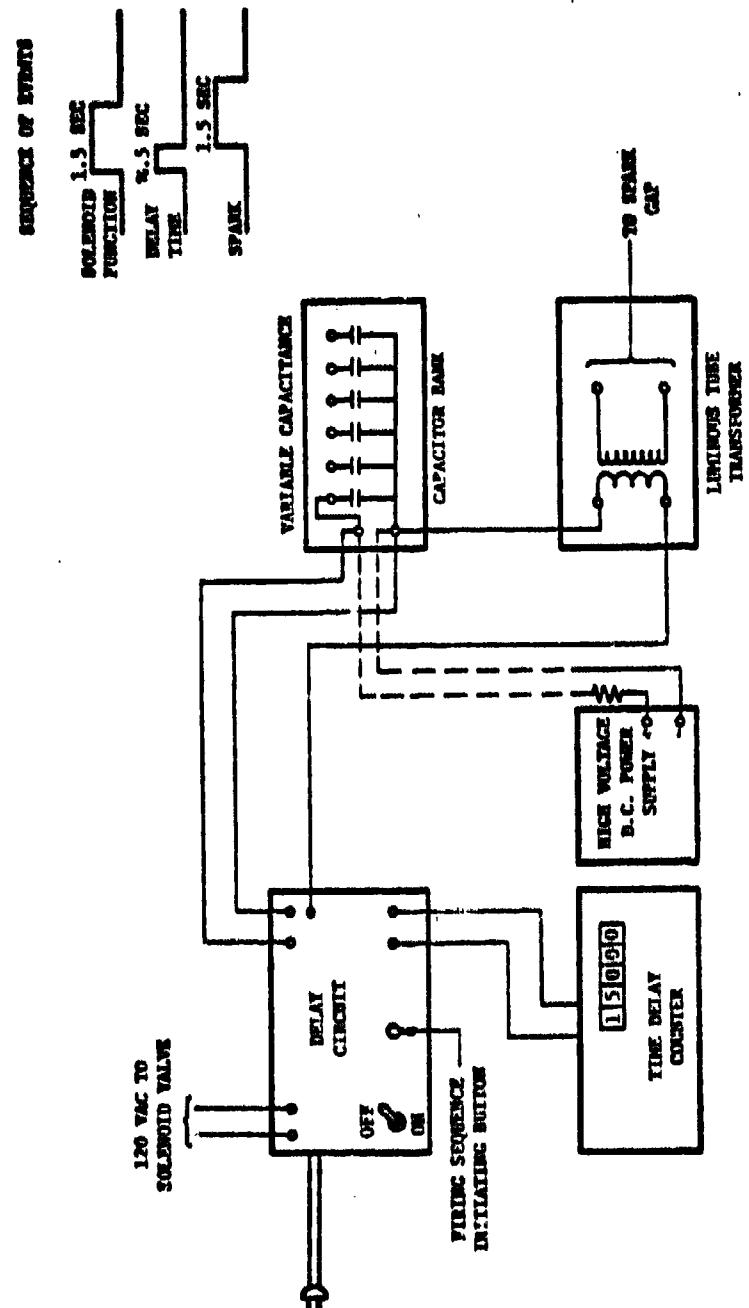


Figure A-4. Electronics System Diagram

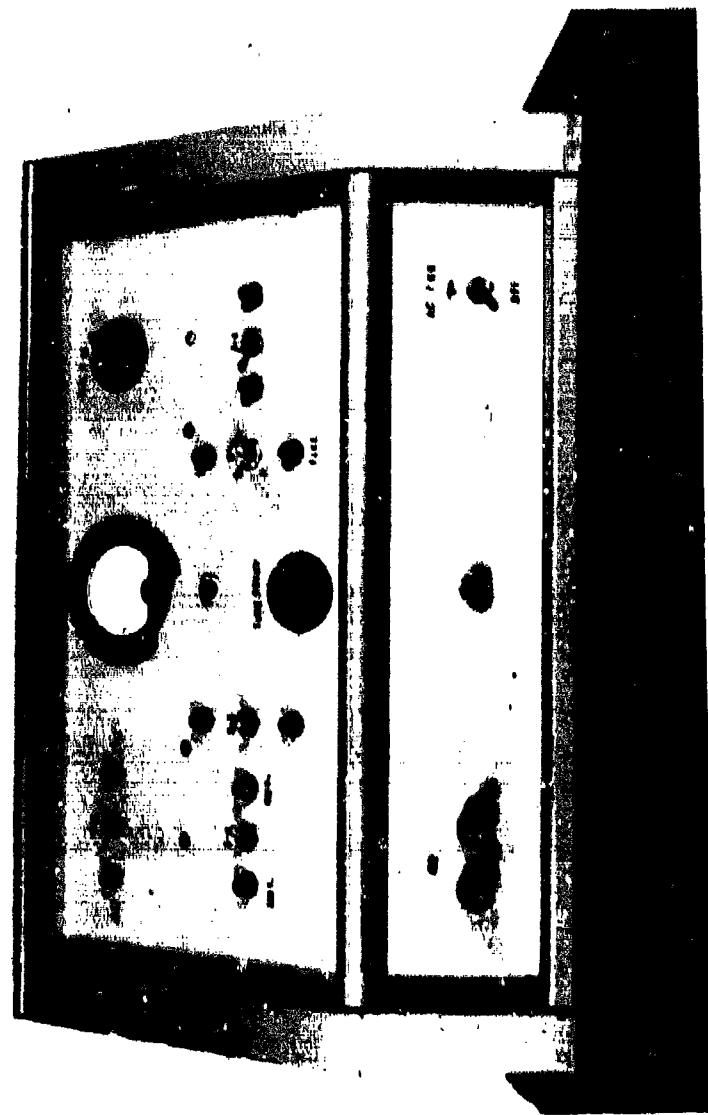


Figure A-5. Electronic control console.

APPENDIX B. USE OF THE PRESSURE-TIME TRACE AS INDICATOR OF FLAME PROPAGATION IN A DUST CLOUD

Visual determination of propagation is not the most accurate technique. The last part of the testing program in Phase II was devoted to the obtaining of pressure traces and attempting to use them along with, or in lieu of, the present visual propagation criteria. A pressure transducer was mounted about halfway up the Lucite tube by drilling a hole into the wall. The pressure-time trace was recorded and photographed through the use of a biomation unit. A biomation unit is essentially an oscilloscope capable of recording a portion of a trace. The trace begins with the release of air from the air reservoir. It was seen that the release of air from the reservoir does not effect a pressure rise. Later tests had the pressure-time trace begin on the spark, since no significant pressure contribution occurred from the air reservoir. Figure B-1 shows the pressure traces for Tests 127 and 128. Propagation (the diaphragm burst) occurs in Test 127, while no reaction occurred in Test 128. Tests 127 and 128 are plotted on the explosibility curve of Figure 12 and correspond to filmed tests 86 and 88, respectively. Note that for Test 127, a peak pressure of about $3,234.3 \text{ kg/m}^2$ occurred at the instant the diaphragm ruptured.

Figure B-2 shows the pressure traces of Tests 141, 142 and 143. All three tests had the same dust concentration, and in all three tests, the diaphragm ruptured thus satisfying the criteria for propagation. Note, however, the difference in the rate of pressure rise in the tests. The rate is highest with the lowest ignition energy.

Unfortunately, no pressure traces were obtained in which propagation nominally occurred (for example, a 10.2-cm flame). The value of the pressure trace would be shown in these nominal cases. However, as stated earlier, the reaction is usually violent (detonation) or minimal (localized flame around gap).

APPENDIX C. PHASE II TEST DATA

Comprehensive data for all 146 tests performed during Phase II of this program (aforementioned in text) are available in attached Appendix Table C-1.

Table C-1 Comprehensive Phase II test data

Test No.	Propellant mass (mg)	Propellant size "a"	Moisture/solvent content	Solution	Temp. °C	Delay time (ns)	Suspension pressure (kPa)	Capacitance (nf)	Voltage (v)	Energy (J)	Result
8/19/77 1	600	200	dry	-	27.5	512	0.318	68.9	50	100	No flame observed (H-1)
8/19/77 2	600	-	200	dry	-	27.5	508	0.318	68.9	100	100
8/19/77 3	600	200	dry	-	29.0	510	0.318	68.9	75	100	Load detonation
8/19/77 4	600	200	dry	-	29.0	511	0.318	68.9	60	100	0.35
8/19/77 5	600	200	dry	-	29.5	510	0.318	68.9	50	100	0.30
8/19/77 6	600	200	dry	-	30.0	508	0.318	68.9	40	100	Slightly less than Tests #5 and #6, but still a good flame
8/19/77 7	600	200	dry	-	31.0	511	0.318	62.9	30	100	Burned with more intensity than Test #5
8/19/77 8	600	200	dry	-	31.0	511	0.318	62.9	30	100	No flame
8/19/77 9	600	200	dry	-	31.0	507	0.318	62.9	30	100	0.15
8/19/77 10	300	200	dry	-	36.0	511	0.318	68.9	30	100	Burned well - not too intense
8/20/77 11	300	200	dry	-	36.0	508	0.318	68.9	50	100	0.15
8/22/77 12	300	200	dry	-	31.5	506	0.318	62.9	70	100	0.25
8/22/77 13	300	200	dry	-	35.0	509	0.318	68.9	80	100	0.35
8/22/77 14	300	200	dry	-	35.5	510	0.318	63.9	60	100	0.30

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture/solvent content	Solution	Temp. °C	Spark time (sec)	Suspension pressure (psi)	Capacitance (μF)	Voltage (V)	Energy (J)	Result
8/23/77	15	300	200	dry	-	36.0	510	0.318	68.9	65	100	0.325 30% propelled, burned, tube partially filled with flame
8/23/77	16	150	200	dry	-	31.0	503	0.318	68.9	70	100	0.35 Spark at gap - no flame at all
8/23/77	17	150	200	dry	-	32.5	507	0.318	68.9	100	100	0.50 Spark at gap - no flame at all
8/23/77	18	150	200	dry	-	34.5	505	0.318	68.9	10	400	0.60 Spark at gap - no flame at all
8/23/77	19	150	200	dry	-	36.0	502	0.318	68.9	14	400	1.12 Spark at gap - no flame
8/23/77	20	150	200	dry	-	37.0	506	0.318	68.9	20	400	1.68 Spark at gap - no flame
8/24/77	21	150	200	dry	-	31.0	505	0.318	68.9	30	400	2.40 Spark at gap - no flame
8/24/77	22	150	200	dry	-	32.5	507	0.318	68.9	75	400	6.00 Spark at gap - small 1.27-cm lateral flame along electrode
8/24/77	23	150	200	dry	-	33.0	506	0.318	68.9	100	400	2.20 Spark at gap - small 1.27-cm lateral flame along electrode
8/24/77	24	200	200	dry	-	33.5	507	0.318	68.9	-	100	0.50 Spark at gap - no flame
8/24/77	25	200	200	dry	-	33.5	505	0.318	68.9	14	400	0.12 Spark at gap - no flame
8/24/77	26	200	200	dry	-	34.5	507	0.218	68.9	25	400	2.60 Spark at gap - small 1.27-cm lateral flame along electrode

Table C-1 Comprehensive Phase II test data (cont'd)

Test No.	Propellant mass (mg)	Propellant size no.	Moisture/solvent content	Solution	Temp. °C	Delay time (ms)	Spark gap (cm)	Suspension pressure (in. Hg)	Capacitance (uf)	Voltage (v)	Energy (J)	Result
8/24/77 27	200	200	dry	-	35.0	507	0.318	68.1	50	400	4.00	Spark at gap - small 1.27-cm lateral flame along electrode
8/24/77 28	200	200	dry	-	35.0	505	0.318	63.9	100	400	8.00	Spark at gap - small 1.27-cm lateral flame along electrode
8/25/77 29	250	200	dry	-	30.5	504	0.318	62.9	100	650	Spark at gap - no flame	
8/25/77 30	250	200	dry	-	21.5	507	0.318	62.9	14	400	1.12	Spark at gap - small 1.27-cm lateral flame along electrode
8/25/77 31	250	200	dry	-	31.5	506	0.318	62.9	75	400	2.06	Spark at gap - small 1.27-cm lateral flame along electrode
8/25/77 32	250	200	dry	-	31.5	506	0.318	62.9	50	400	4.00	Spark at gap - small 1.27-cm lateral flame along electrode
8/25/77 33	250	200	dry	-	33.5	498	0.318	62.1	100	400	8.00	Spark at gap - small 1.27-cm lateral flame along electrode
8/25/77 34	450	200	dry	-	34.0	502	0.318	62.9	50	100	0.25	Spark at gap - no flame
8/25/77 35	450	200	dry	-	30.5	501	0.318	68.9	100	100	0.50	Spark at gap - no flame - reserve pressure 34.5 kPa

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture solvent content	Temp. (°C)	Solution	Delay time (rs)		Start gap (cm)		Suspens. on pressure (kPa)		Capacitance (μF)		Voltage (V)		Energy (J)		Result
							Start	End	Start	End	Start	End	Start	End	Start	End	Start	End	
8/26/77	36	150	200	dry	-	-	31.5	501	0.319	5e-4	100	400	8.00	8.00	Sparks at gap - small 1.27-cm lateral flame along electrode - Before Test #36, check valve was adjusted for "0" gauge pressure without rupture diaphragm. After test, 13.8 kPa residual reserve pressure with diaphragm.				
8/26/77	37	200	200	dry	-	-	32.5	400	0.320	5e-4	25	400	2.80	2.80	Sparks at gap - small 1.27-cm lateral flame along electrode				
8/26/77	38	200	200	dry	-	-	33.5	400	0.316	6e-4	50	400	4.00	4.00	Sparks at gap - small 1.27-cm lateral flame along electrode - slightly more				
8/26/77	39	200	200	dry	-	-	34.0	502	0.312	6e-4	-	-	-	-	400	400	Sparks at gap - small 1.27-cm lateral flame along electrode - about same as above		
8/31/77	40	300	200	dry	-	-	30.0	511	0.319	6e-4	76	190	0.35	0.35	Sparks at gap - no flame at all				
8/31/77	41	300	200	dry	-	-	30.0	501	0.318	6e-4	100	100	0.50	0.50	detonation? - filter paper discharge rupture - no flame				
8/31/77	42	300	200	dry	-	-	30.0	502	0.318	6e-4	80	100	0.40	0.40	Sparks at gap - no flame				
8/31/77	43	300	200	dry	-	-	30.5	503	0.318	6e-4	90	100	0.45	0.45	Sparks at gap - no flame				

Table C-1 Comprehensive Phase II test data (cont'd)

Test No.	Fuel mass (mg)	Propellant size no.	Moisture/ solvent content	Solution	Temp. of	Relay time (ms)	Spark gap (cm)	Suspension pressure (kPa)	Capacitance (nf)	Voltage (v)	Energy (j)	Result	
												9/1/77	44
9/1/77	45	300	200	dry	-	24.5	500	0.318	32.7	60	100	0.30	Detonation? - diaphragm rupture - no flame
9/1/77	46	300	200	dry	-	27.5	502	0.318	62.9	100	100	0.50	Spark at gap - no flame
9/1/77	47	600	200	dry	-	27.5	502	0.318	62.9	75	100	0.375	Detonation? - diaphragm rupture - no flame
9/1/77	48	600	150	dry	-	28.0	502	0.318	68.9	50	100	0.25	Spark at gap - no flame
9/2/77	49	600	150	dry	-	24.5	497	0.318	62.9	100	100	0.50	Fire in tube at 5.08-6.35 cm above and below electrode followed by rupture of diaphragm - over 1/2 burned
9/2/77	50	600	150	dry	-	25.5	512	0.318	62.9	90	100	0.45	Same results as Test #49, except fire in tube not quite as large - more residue in tube
9/2/77	51	600	150	dry	-	26.0	499	0.318	62.9	80	100	0.40	Tube partially filled with flame - diaphragm ruptured as before
9/2/77	52	600	150	dry	-	29.5	502	0.318	68.9	70	100	0.35	Same basic results as Test #51
9/2/77	53	600	150	dry	-	31.0	503	0.318	68.9	60	100	0.30	Same basic results as Test #51

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture/solvent content	Temp. $^{\circ}\text{C}$	Relay time (hrs)	Spark gap (cm)	Suspension pressure (kPa)	Capacitance (nf)	Voltage (v)	Energy (J)	Result	
												diaphragm ruptured	diaphragm ruptured
9/6/77	54	600	150	dry	-	23.0	497	0.318	68.9	50	100	0.25	Spark at gap - tube partially filled with flame - diaphragm ruptured
9/6/77	55	600	150	dry	-	24.0	498	0.318	68.9	40	100	0.20	Spark at gap - no flame
9/6/77	56	600	150	dry	-	24.5	498	0.318	68.9	45	100	2.225	Spark at gap - tube partially filled with flame - diaphragm ruptured
9/6/77	57	300	150	dry	-	24.5	497	0.318	68.9	60	100	0.30	Spark at gap - tube partially filled with flame - diaphragm ruptured
9/6/77	58	300	150	dry	-	25.5	499	0.318	68.9	50	100	0.25	Spark at gap - no flame
9/6/77	59	300	150	dry	-	25.5	499	0.318	68.9	55	100	0.275	Tube partially filled with flame - diaphragm ruptured
9/6/77	60	150	150	dry	-	27.0	498	0.318	68.9	100	100	0.50	Spark at gap - no flame
9/6/77	61	150	150	dry	-	27.0	500	0.318	68.9	50	400	4.00	Spark at gap - no flame
9/7/77	62	150	150	dry	-	23.0	497	0.318	68.9	100	400	8.00	Spark at gap - no flame
9/7/77	63	450	150	dry	-	25.0	511	0.318	68.9	50	100	0.25	Fire filled a large portion of tube - diaphragm ruptured
9/7/77	64	450	150	dry	-	25.5	499	0.318	68.9	40	100	0.20	Fire partially filled tube - diaphragm ruptured

Table C-1 Comprehensive Phase II test data (cont'd)

Test No.	Propellant mass [mg]	Propellant size no.	Moisture/solvent content	Leap-off solution	Relay time [ms]	Spark gap [cm]	Suspension pressure [kPa]	Capacitance [nf]	Voltage [v]	Energy [J]	Result		
9/7/77	65	450	150	dry	-	26.0	500	0.318	68.9	30	100	0.15	Spark at gap - no flame
9/7/77	66	450	150	dry	-	27.0	500	0.318	68.9	35	200	0.175	Spark at gap - tube partially filled with flame - diaphragm ruptured
9/12/77	67	150	150	wet	water	20.0	496	0.318	68.9	100	100	0.5	Spark at gap - no flame
9/12/77	68	200	150	wet	water	19.0	493	0.318	68.9	100	400	8.0	Spark at gap - no flame
9/12/77	69	200	150	wet	water	21.0	496	0.328	62.9	100	100	9.0	Spark at gap - small flame around gap (1.27 cm)
9/12/77	70	300	150	wet	water	22.0	496	0.318	68.9	100	400	8.0	Spark at gap - small flame along electrode (2.54 cm)
9/12/77	71	450	150	wet	water	23.0	496	0.318	68.9	100	400	8.0	Spark at gap - small flame around gap (1.27 cm)
9/13/77	72	150	150	10%	ethyl al.	22.5	497	0.318	68.9	10	400	0.8	Spark at gap - no flame (0.64 cm)
9/14/77	73	150	160	10%	ethyl al.	23.0	498	0.318	68.9	25	400	2.60	Spark at gap - small flame around gap (0.64 cm)
9/14/77	74	150	140	10%	ethyl al.	24.0	496	0.318	68.9	100	400	8.00	Spark at gap - small flame around gap (1.27 cm)
9/14/77	75	300	140	10%	ethyl al.	25.5	500	0.318	68.9	160	100	0.50	Spark at gap - small flame around gap (1.27 cm) then small running flame along 1 electrode

Table C-1 Comprehensive Phase II test data (cont'd)

<u>Test No.</u>	<u>Propellant mass (mg)</u>	<u>Propellant size no.</u>	<u>Moisture/ solvent content</u>	<u>Solution</u>	<u>Temp. $^{\circ}\text{C}$</u>	<u>Delay time (ns)</u>	<u>Spark gap (cm)</u>	<u>Suspension pressure (kPa)</u>	<u>Capacitance (uf)</u>	<u>Voltage (v)</u>	<u>Energy (J)</u>	<u>Result</u>
9/14/77 76	300	140	10%	ethyl al.	24.0	496	0.318	68.9	25	400	2.00	Spark at gap - small flame around gap (1.27 cm)
9/14/77 77	300	140	10%	ethyl al.	21.5	496	0.318	68.9	100	400	8.00	Spark at gap - small flame around gap (1.27 cm) and along electrode (2.54 cm)
9/14/77 78	450	140	10%	ethyl al.	21.0	494	0.318	68.9	25	400	2.00	Spark at gap - small flame around gap (1.27 cm) and along electrode (2.54 cm)
9/14/77 79	450	140	10%	ethyl al.	25.5	497	0.318	68.9	100	400	8.0	Spark at gap - slightly larger flame around gap seemed to be a little brighter
9/14/77 80	150	200	10%	ethyl al.	24.0	496	0.318	68.9	100	100	0.50	Spark at gap - no flame
9/14/77 81	150	200	10%	ethyl al.	24.0	496	0.318	68.9	25	400	2.00	Spark at gap - small 1.27-cm lateral flame
9/14/77 82	150	200	10%	ethyl al.	24.0	497	0.318	68.9	100	400	8.0	Spark at gap - small 1.27-cm lateral flame
9/15/77 83	450	140	dry	-	24.0	496	0.318	68.9	50	100	0.25	Spark at gap - no flame - repeat of Test #63 - 16 mm at 64 fps
9/15/77 84	300	140	dry	-	25.5	498	0.318	68.9	100	100	0.50	Spark at gap - no flame - Tests #57-#59 series - 16 mm at 64 fps

Table C-1 Comprehensive Phase II test data (cont'd)

Test No.	Propellant mass (mg)	Propellant size no.	Moisture/ solvent content	Solvent	Temp. Δt	Delay time [sec]	Suspension pressure (kPa)	Capacitance (nf)	Voltage 'V' (V)	Energy (J)	Result
9/15/77 85	600	140	dry	-	28.0	500	0.635	58.9	100	~8.00	Spark at gap - flame developed from spark up (7.62 cm), then developed from bottom as dust settles - no detonation but diaphragm slightly torn, 16 mm
9/15/77 86	600	140	dry	-	29.0	500	0.635	68.9	25	400	2.00
9/15/77 87	600	140	dry	-	27.5	500	0.635	68.9	100	100	Spark at gap - flame filled all of tube, then diaphragm ruptured - 16 mm
9/15/77 88	600	140	dry	-	27.5	498	0.635	68.9	50	100	0.25
9/15/77 89	300	200	10% ethyl al.	26.0	499	0.318	68.9	100	100	0.50	Spark at gap - no flame
9/15/77 90	300	200	10% ethyl al.	26.0	498	0.318	68.9	25	400	2.00	Spark at gap - seal 1.27-cm lateral flame
9/15/77 91	300	200	10% ethyl al.	25.5	497	0.318	68.9	100	400	8.0	Spark at gap - seal 2.54-cm lateral flame
9/16/77 92	450	200	10% ethyl al.	22.0	497	0.318	68.9	25	400	2.00	Spark at gap - flame streaks from gap up to 5.08 cm above
9/16/77 93	150	140	10% an. ether	21.0	496	0.318	68.9	100	100	0.50	Spark at gap - no flame

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture/solvent content	Solution	Temp. °C	Delay time [ns]	Start gap [cm]	Suspension pressure [bara]	Capacitance [pf]	Voltage (v)	Energy (J)	Result	
													22.0	495
9/16/77	94	150	140	10 ²	an. ether	22.0	495	0.318	68.9	100	400	8.00	Spark at gap - small flame along spark	
9/16/77	95	150	140	10 ²	an. ether	23.0	497	0.318	68.9	100	400	8.00	Spark at gap - small flame along spark	
9/16/77	96	300	140	10 ²	an. ether	24.5	497	0.318	68.9	100	400	8.50	Spark at gap - no flame	
9/16/77	97	300	140	10 ²	an. ether	24.5	498	0.318	68.9	25	400	2.30	Spark at gap - small flame along spark	
9/16/77	98	300	140	10 ²	an. ether	25.0	496	0.318	68.9	100	400	8.00	Spark at gap - small flame around gap	
9/16/77	99	450	160	10 ²	an. ether	24.5	498	0.318	68.9	25	400	2.00	Spark at gap - no flame	
9/19/77	102	450	160	10 ²	an. ether	22.0	496	0.318	68.9	100	400	8.0	Spark at gap - small flame along spark	
9/19/77	101	150	200	10 ²	an. ether	22.5	498	0.318	68.9	100	400	0.50	Spark at gap - no flame	
9/19/77	102	150	200	10 ²	an. ether	22.0	497	0.318	68.9	25	400	2.00	Spark at gap - no flame	
9/19/77	103	150	200	10 ²	an. ether	22.5	497	0.318	68.9	100	400	8.30	Spark at gap - small flame along spark	
9/19/77	104	300	200	10 ²	an. ether	24.0	498	0.318	68.9	100	400	0.50	Spark at gap - no flame	
9/19/77	105	300	200	10 ²	an. ether	23.0	497	0.318	68.9	25	400	2.00	Spark at gap - small flame along gap	

Table C-1 Comprehensive Phase II test data (cont'd)

Propellant mass (mg)	Test No.	Propellant size no.	Moisture/ solvent content	Solution	Temp- QC	Delay time (Sec.)	Susten- sion pressure (10 ³ Pa)	Capacitance (uF)	Voltage (V)	Energy (J)	Result
9/19/77 106	300	200	10%	an. ether	23.0	495	0.318	68.9	100	400	8.00
9/19/77 107	450	200	10%	an. ether	24.0	494	0.318	68.9	25	400	2.00
9/19/77 108	450	200	10%	an. ether	24.5	497	0.318	68.9	100	400	8.00
9/19/77 109	450	140	at 2 min exp time	ether	25.0	497	0.318	68.9	100	400	8.00
9/20/77 110	300	140	dry	ether	26.0	500	0.318	68.9	75	100	0.375
9/20/77 111	300	140	dry	-	27.0	501	0.635	68.9	100	100	0.50
9/20/77 112	450	140	dry	-	27.0	498	0.635	66.9	100	100	0.50
9/20/77 113	450	140	dry	-	27.5	501	0.635	66.9	25	400	2.00
9/20/77 114	-	-	1/4 cc at 1 mm	ether	27.0	498	0.635	68.9	25	400	2.00
9/20/77 115	-	-	12 drops at 1 mm	ether	26.5	498	0.635	68.9	25	400	2.00

Table C-1 Comprehensive Phase II test data (cont'd)

<u>Propellant</u>	<u>Test No.</u>	<u>Propellant mass (mg)</u>	<u>Moisture/ solvent content</u>	<u>Solution size no.</u>	<u>Temp. °C</u>	<u>Relay time (ms)</u>	<u>Spark gap (cm)</u>	<u>Suspension pressure (kPa)</u>	<u>Capacitance (uf)</u>	<u>Voltage (v)</u>	<u>Energy (J)</u>	<u>Result</u>
9/20/77	116	-	-	20 drops at 1 mm	ether	25.5	490	0.635	68.9	25	400	Spark at gap - flame filled tube very quickly, then loud bang with rupture of diaphragm
9/20/77	117	-	-	15 drops at 1 mm	ether	25.5	497	0.635	68.9	25	400	Spark at gap - flame filled tube very quickly, then loud bang with rupture of diaphragm
9/20/77	118	-	-	10 drops at 1 mm	ether	25.5	496	0.635	68.9	25	400	Spark at gap - very slight reaction above part (blue flame) (0.315 cm)
9/20/77	119	-	-	12 drops at 1 mm	ether	25.5	495	0.635	68.9	25	400	Spark at gap - blue flame filled quickly, then loud bang with rupture of diaphragm (spare flame)
9/20/77	120	-	-	10 drops at 1 mm	ether	25.0	496	0.635	68.9	25	400	Spark at gap - small flame along spark
9/20/77	121	450	140	10 drops at 1 mm	ether	25.0	495	0.635	68.9	25	400	Spark at gap - flame filled tube quickly, then loud bang with rupture of diaphragm
9/20/77	122	304	140	10 drops at 1 mm	ether	25.0	495	0.635	68.9	25	400	Spark at gap - flame filled tube quickly, then loud bang with rupture of diaphragm

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture/ solvent content	Solvent	Temp. at 1 mm	Suspension pressure		Capacitance (uf)	Voltage (V)	Energy (J)	Result
							Time (ms)	Gap (cm)				
9/20/77	123	150	140	10 drops at 1 mm	ether	25.0	498	0.635	62.3	25	460	2.00
9/20/77	124	150	140	10 drops at 1 mm	ether	24.5	495	0.476	62.9	100	100	0.50
9/21/77	125	600	140	dry	-	22.0	500	0.635	62.9	25	400	2.00
9/21/77	126	600	140	dry	-	23.0	503	0.635	62.9	25	400	2.00
9/21/77	127	600	140	dry	-	23.0	473	0.635	68.9	25	400	2.00
9/21/77	128	600	140	dry	-	24.0	492	0.635	68.9	50	100	0.25
9/21/77	129	600	140	dry	-	24.0	492	0.635	68.5	75	100	0.375
9/22/77	130	150	140	10 drops at 1 mm	ether	26.0	490	0.318	68.9	50	100	0.25

Table C-1 Comprehensive Phase II test data (cont'd)

Date	Test No.	Propellant mass (mg)	Propellant size no.	Moisture/ solvent content	Solution	Delay time (ms)	Spark gap (cm)	Suspension pressure (kPa)	Capacitance (μF)	Voltage (V)	Energy (J)	Result
9/22/77	131	-	-	15 drops at 1 sec	ether	21.0	0.635	66.5	25	90	2.0x	Spark at gap - sparse blue flame filled tube quickly, then loud bang with rupture of diaphragm
9/22/77	132	-	-	10 drops at 1 sec	ether	22.0	0.576	68.9	100	100	0.50	Spark at gap - small blue flame at gap area; developed late, then bang with rupture of diaphragm
9/22/77	133	-	-	5 drops at 1 sec	ether	23.0	0.476	69.9	100	100	0.50	Spark at gap - no flame
9/22/77	134	150	140	5 drops at 1 sec	ether	24.5	0.424	65.4	200	100	0.50	Spark at gap - small 3.72-cm flame at and above gap (diaphragm intact)
9/22/77	135	-	-	8 drops at 1 sec	ether	24.5	0.476	68.7	100	100	0.50	Spark at gap - no flame
9/22/77	136	150	140	8 drops at 1 sec	ether	25.5	0.376	63.5	100	100	0.50	Spark at gap - flame filled tube, other end being with rupture of diaphragm
9/22/77	137	150	140	8 drops at 1 sec	ether	24.0	0.388	63.5	50	100	0.25	Spark at gap - 6.35-cm flame streak (blue) from gap diagonally up to tube wall; diaphragm intact
9/22/77	138	-	-	15 drops at 1 sec	ether	24.0	0.635	68.~	25	400	2.00	Spark at gap - flame filled tube very quickly, then loud bang with rupture of diaphragm

Table C-1 Comprehensive Phase II test data (cont'd)

Expt.	Test No.	Propellant mass (mg)	Propellant size no.	Posture/solvent content	Temp. (°C)	Relay time [sec]	Spark gap (cm)	Sustained pressure (Pa)	Capacitance (fF)	Voltage (V)	Energy (J)	Result
9/22/77	139	150	140	15 drops at 1 mm	ether	24.0	459	0.635	68.9	25	400	2.00
9/22/77	140	150	150	16 drops at 1 mm	ether	24.0	456	0.310	68.9	25	100	0.125
9/22/77	141	450	140	dry	ether	25.5	497	0.635	68.9	25	400	2.00
9/22/77	142	450	140	dry	ether	24.5	495	0.635	68.9	12	400	1.04
9/22/77	143	450	140	dry	ether	25.0	497	0.476	68.9	100	100	0.50
9/23/77	144	300	140	8 drops at 1 mm	ether	22.0	493	0.316	68.9	40	100	5.20
9/23/77	145	450	140	8 drops at 1 mm	ether	22.0	497	0.318	68.9	25	100	0.125
9/23/77	146	600	140	8 drops at 1 mm	ether	24.0	496	0.310	68.9	30	100	0.15

Note: diaphragm made from laboratory filter paper, Grade 615.

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